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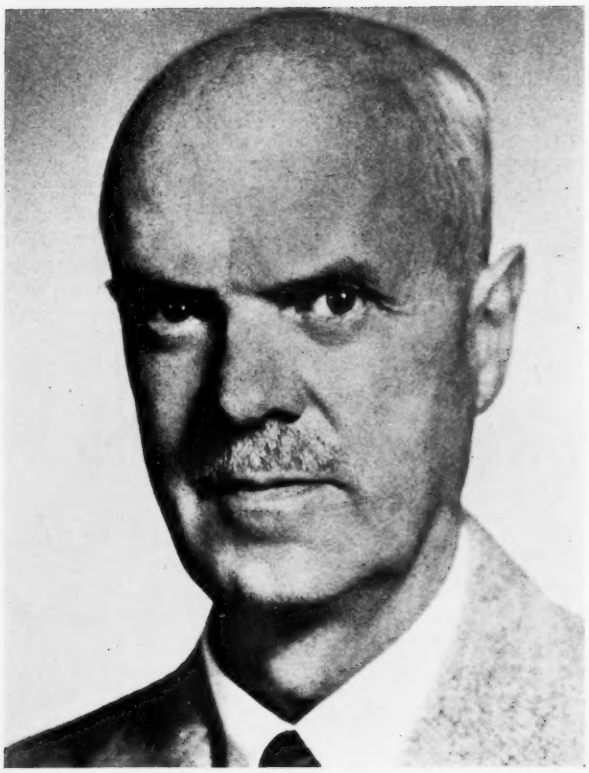

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PAPERS FOR THE MEETING IN SWEDEN

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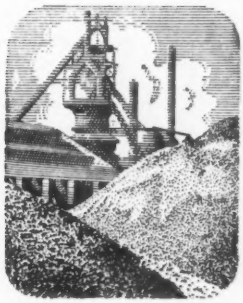

Iron and Steel Making Processes Used in Sweden

By Sven Fornander

SYNOPSIS

After a historical introduction and a short account of the raw material situation, a description is given of the various processes used in Sweden for the making of pig iron, sponge iron, and ingot steel. Emphasis is laid on the characteristic features of Swedish technology compared with foreign practice, which are due mainly to Sweden's scarcity of fossil fuel and its relatively large resources of hydro-electric power. 936

HISTORICAL SURVEY

IRONMAKING has been known in Sweden for about 2000 years.[1] The raw material first used was bog ore, and therefore the ironmaking was localized mainly in the southern part of the country (the province of Småland), where such ore is abundant. Not until the twelfth century was rock ore used to any considerable extent, and this change caused the geographical centre of ironmaking to be moved to the ore district of Central Sweden.

At first the ore was reduced in shallow pits in the ground, which by degrees were developed into low furnaces. Gradually blast was introduced. Owing to the low working temperature of the furnaces, the product obtained—the so-called 'osmund iron'—was forgeable. Export of osmund iron was begun in the thirteenth century but, according to present-day standards, the quantities were small; as late as the sixteenth century, when the export of this type of iron reached its highest level, it never exceeded 4500 tons per year.[2] The osmund iron was delivered in the shape of small lumps, packed in wooden casks.

It is not possible to state exactly when the blast-furnace process was first applied in Sweden. From the middle of the fifteenth century, however, the use of blast-furnaces has been definitely established. In the sixteenth century, refining of the pig iron in hearths was begun. After removal of the slag by forging, the wrought iron was hammered into bars. For several hundred years after the introduction of the blast-furnace process the technical development did not involve any fundamentally new type of progress, but was characterized by attempts to increase the efficiency of blast-furnace practice and to improve the hearth refining process. In the seventeenth century production was started on a larger scale, and it was at this time that the typical Swedish ironworks were established. The iron-masters became an important class of society of high social standing.

For a long time, from the latter half of the seventeenth to the end of the eighteenth century, Swedish iron production was dominant in Europe. This was because Sweden—to a greater extent than any other country—had access to all the production factors necessary at that time for the making of iron on a larger scale. There was an abundant supply of rich ores with low contents of harmful impurities. The only type of fuel that could be used in ironmaking, *i.e.*, charcoal, could be produced in large quantities from wood out of vast forests. That large quantities of fuel were needed can be seen from information given by Jernkontoret's outstanding metallurgist, Sven Rinman, in his great work " Bergwerks Lexicon ", in which he collected the metallurgical knowledge of his time (the 1780's). The consumption of charcoal for pig-iron production in the blast-furnace was about 100 hl. per ton,[3] corresponding to about 140% by weight of the pig iron produced. For the refining of the pig iron and the final forging of the wrought iron to rods the charcoal consumption was of the order of 250

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Mr. Fornander is Director of Research of Jernkontoret.

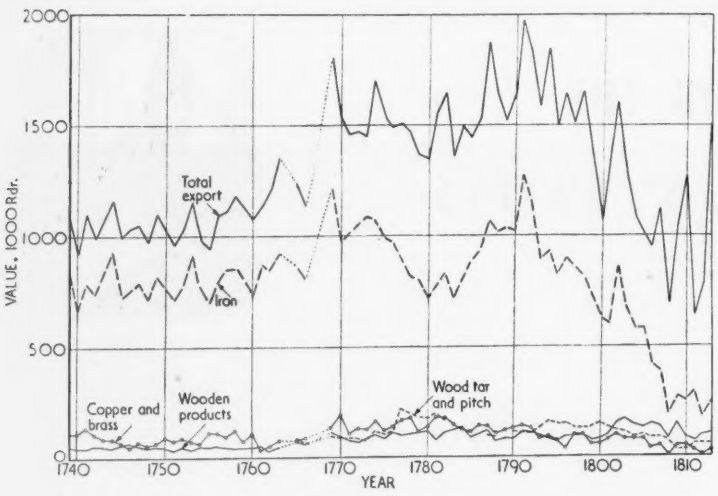

Fig. 1—The value of Swedish exports for the period 1738–1813

hl. per ton,[4] and the fuel requirement for the production of 1 ton of rod iron from ore amounted to the enormous quantity of 400 hl., *i.e.*, about 5 tons. As a similar technique was applied abroad, it was especially in regard to fuel that the large forests of Sweden gave the country a unique position compared with almost all other countries, where at this time the scarcity of charcoal had become very serious. Also, Sweden had large reserves of power, the third important production factor for ironmaking. There were many waterfalls of suitable size, and they were not far from ore or fuel.

During this period Sweden was the largest iron-producing country in Europe ; at certain times the country provided no less than 30–35% of world production. According to modern standards, however, the quantities were quite small : at the beginning of the eighteenth century the annual production of wrought iron in Sweden amounted to about 40,000 tons, and towards the end of the century to about 60,000 tons. About 80–85% of the production was exported. The dominating rôle of the iron export in the foreign trade of Sweden during the period 1739–1813 is shown in Fig. 1, which is taken from the work of Heckscher.[5] The main importing country was England, which usually absorbed at least half and sometimes as much as two-thirds of the Swedish iron export. During certain years the Swedish export to England was as great as the home production of that country. Not until the 1730's did Swedish iron exports meet serious competition. This came from Russia, which had a good supply of raw materials and where the iron industry had been organized at the beginning of the eighteenth century. Soon, however, Sweden and Russia together could hardly satisfy the growing demand.

It was not only in regard to quantity that Sweden held a unique position. Much was done to keep the quality at a high level. Particularly well-known for its quality was the so-called 'Orground iron' (Öregrund iron), which was made at Leufsta, Osterby, and Söderfors from Dannemora ore according to the Walloon hearth refining process. The Orground iron had a comparatively high carbon content, and therefore was specially suitable as a raw material for the production of hard steel. The recognition of the importance of quality by the leading ironmasters became an invaluable asset when the industry met its great crisis at the beginning of the nineteenth century.

The important inventions made in England during the eighteenth century revolutionized ironmaking technically and commercially. Coke was first used in blast-furnaces in the 1730's (A. Darby), and crucible steelmaking was introduced in the 1740's (B. Huntsman). The most important new method was the puddling process, developed in the 1780's by Henry Cort, the product obtained being rolled in rolling mills to the required dimensions. The introduction of this new technique, based on fossil fuel, transferred the leading position from Sweden to England which, at the beginning of the nineteenth century, became the largest iron producer.

That the Swedish iron industry succeeded in surviving its difficult crisis was due mainly to three circumstances. The first was the industrial revolution that originated in England and later spread to other countries, and led to a highly increased demand for iron and also for steel. The second was that owing to its high quality Swedish iron had a very good reputation on foreign markets. The third factor was the ability of the Swedish industry to adapt itself gradually to the new situation. Production was directed to high-grade iron, *i.e.*, wrought iron produced with charcoal, which was superior to the puddled iron as raw material for blacksmiths' work and steelmaking. Development was slow, and several attempts were

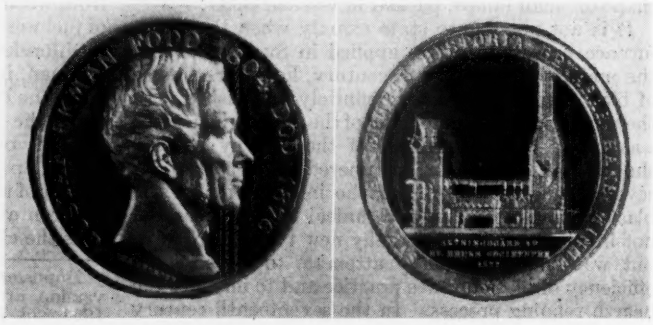

Fig. 2—Medal struck by Jernkontoret in memory of Gustaf Ekman

made along different lines before the process was perfected. An interesting account of the important part played by Jernkontoret in this connection has recently been prepared.[6] The most important individual contribution to the success of the new process was that of Gustaf Ekman, who introduced from England a more efficient hearth refining method, the Lancashire process, which he improved. He also designed heating furnaces, thus enabling the product to be shaped by rolling. In 1877 Jernkontoret struck a medal in memory of Ekman (*see* Fig. 2).

The ingot steel methods invented abroad were soon introduced in Sweden. The Bessemer process had already been taken up in Sweden in 1858 by G. F. Göransson, who founded the Sandviken Steel Works a few years later to exploit the method. The first open-hearth heat in Sweden was made at Munkfors in 1867. An early Swedish contribution to the development of electric steel furnaces was made by F. A. Kjellin, who built the first low-frequency induction furnace in 1899.

During the twentieth century an electric furnace for pig-iron making, the so-called Elektrometall furnace, was developed in Sweden, and the problem of sponge-iron production was solved technically and commercially. Two methods, those of Sieurin (the Höganäs method) and of Wiberg, are now in operation. An attempt is made in this paper to give a picture of the latest development of Swedish iron- and steel-making, and to point out characteristic features of the methods used, in comparison with the application in other countries.

A number of general surveys of iron- and steel-making in Sweden are to be found in the literature. Reference is made here only to Åselius,[7] who has discussed the subject mainly from an economic stand-

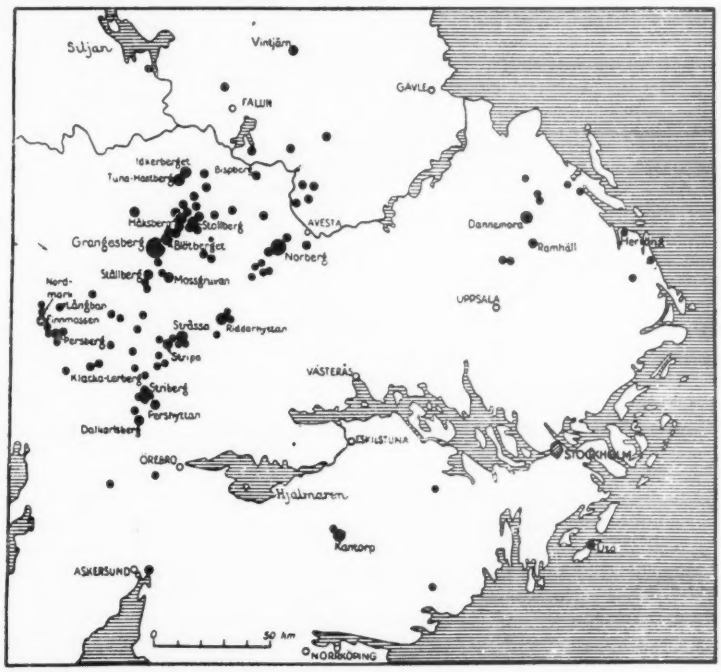

Fig. 3—The iron ore resources of Central Sweden. (After Geijer and Magnusson[11])

point, and to Kalling,[8] whose treatment deals mainly with technical aspects.

RAW MATERIALS

Iron Ore

The Swedish iron ore deposits are situated mainly in two areas–one in Central Sweden and the other in Lapland (*see* Figs. 3 and 4). The Central Swedish iron ore district, often called Bergslagen, is roughly in the shape of a horseshoe, running from Dannemora in Upland, over Grängesberg in Dalarna and Persberg in Värmland, to Kantorp and Utö in Södermanland. The Lapland iron ores, of which the most important are found in the Kiruna and Gellivare ore fields, are situated to the north of the arctic circle.

The geology and structure of the ores have been carefully investigated and are described in detail by Geijer and Magnusson.[9, 10] The extent of the ore reserves is fairly well known. According to the latest estimate made by these authors in 1952,[11] the total resources of lump ore and concentrates should amount

Table I

SWEDISH IRON ORE RESOURCES ACCORDING TO GEIJER AND MAGNUSSON[11]

	Lump Ore and Concentrates, tons × 10^6	Iron Contained, tons × 10^6
Central Sweden	**265**	**151**
Lapland	**2413**	**1532**
Titaniferous iron ores	**213**	**66**
Total	**2891**	**1749**

to about 2900 million metric tons, containing about 1750 million tons of iron (*see* Table I). Most of this ore quantity consists of lump ore rich in phosphorus and with an iron content of 60% or more. The Lapland deposits are considerably larger than those of Central Sweden.

Swedish iron ore mining has in later years been considerably improved and extended.[12–15] In 1953 the total ore production amounted to about 17·1 million tons, of which the main part, about 14·6 million tons, was exported. At present, Sweden's iron ore export is the largest in the world. During the years since the war about one-third of the iron ore imported by Great Britain has been of Swedish origin.[16]

The ores from Central Sweden that form the basis for the production of high-grade steel consist of hard magnetites or hematites, which are rather lean in the mined state, generally with iron contents of between 30 and 40%. As a rule they are dressed magnetically or by wet gravity methods, or by a combination of both these processes, and the concentrates obtained usually contain 60–65% of iron and low amounts of phosphorus. Typical analyses of ores as mined and of concentrates are given in Table II, which also contains two analyses of export ore from Kiruna and Gellivare. The figures given show that the concentrates obtained from the lean ores constitute an exceedingly good raw material for pig-iron production. For the making of sponge iron a concentrate with a still higher iron content is desired ; an example is included in Table II.

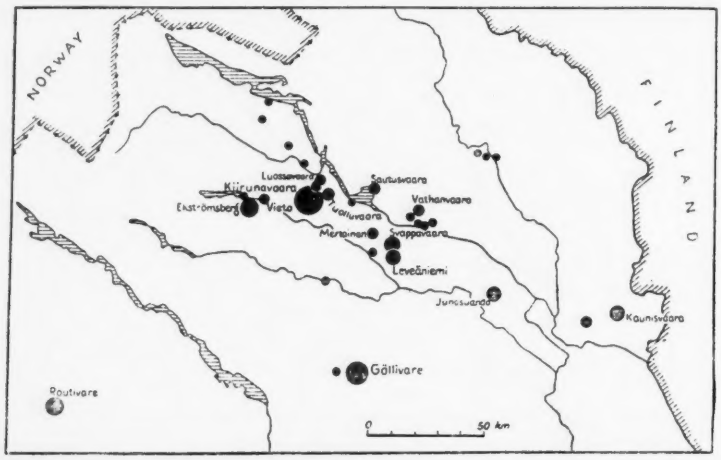

Fig. 4—The iron ore resources of Northern Sweden. (After Geijer and Magnusson[11])

The development of the beneficiation technique[17] has been strongly furthered by the research work that the Swedish Mining Association (Svenska Gruvföreningen) is carrying out in co-operation with Jernkontoret, and by the establishment of a well-equipped laboratory for mineral beneficiation at the Royal Institute of Technology, Stockholm.

Fuel and Electrical Power

As already mentioned, Sweden is almost entirely devoid of fossil fuel. The iron and steel industry is therefore dependent on imported fuel, especially coke,

Table II

COMPOSITION OF IRON ORES AND CONCENTRATES

Analysis, %	Bondgruvan Ore			Persberg Ore		Bodås Ore		Kiruna 'D' Lump Ore	Gellivare 'A10' Concentrate
	As Mined	Magnetite Concentrate*	Hematite Concentrate*	As mined	Concentrate†	Magnetically Sorted	Concentrate†		
SiO_2	35·2	6·8	1·2	29·0	4·2	15·3	2·6	2·7	0·5
Al_2O_3	1·6	0·5	0·3	3·9	1·1	2·9	0·3	0·5	0·3
Fe_2O_3	40·3	64·1	94·3	28·7	65·5	46·6	64·3	59·0	69·1
FeO	8·2	24·7	1·5	15·6	25·5	25·5	32·3	24·3	29·7
MnO	0·08	0·05	0·02	0·3	0·2	0·5	0·4	0·1	0·1
CaO	4·7	1·2	0·9	9·9	1·5	3·5	0·7	6·6	0·2
MgO	6·1	0·8	0·01	11·7	1·8	1·0	0·5	1·2	0·3
P_2O_5	0·038	0·020	0·018	0·014	0·009	0·032	0·006	4·48	0·011
Fe	34·5	64·0	67·1	32·2	65·6	52·4	70·1	60·1	71·4
P	0·016	0·009	0·008	0·006	0·004	0·014	0·003	1·96	0·025
S	0·13	0·027	0·29	0·006	0·005	2·6	0·30	0·016	0·010

* For the production of pig iron † For the production of sponge iron

oil, and coal. Blast-furnace coke is produced from coal only to a limited extent.

Statistical data on the consumption of different types of fuel are available over a fairly long period.[18] Figure 5 shows the relative importance of the different fuels used in Swedish iron and steel works during the years 1930 to 1952.* At the beginning of the century charcoal was still the only reducing agent used in the making of pig iron, and was then the most important fuel. Since then it has gradually become of less importance, and in 1952 only about 10% of the total fuel consumption consisted of charcoal. The importance of coke has increased rapidly in later years : half of the total fuel quantity consumed in 1952 was coke and coke breeze. As in the steel industries of the large industrial countries, the use of fuel oil has increased considerably in later years whereas the use of coal, relatively speaking, has decreased. The open-hearth furnaces have to a large extent been rebuilt for burning heavy fuel oils. This is also the case with the reheating furnaces in rolling mills and forging shops. In 1952 no less than one-quarter of the fuel consumption was of oil.

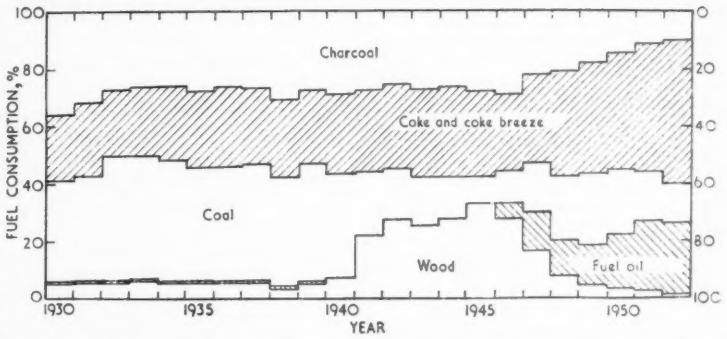

Fig. 5—Consumption of various types of fuel in the Swedish iron and steel industry (relative figures) in the period 1930–1952

During the last war, coal could not be imported to the same extent as it had been earlier. The fuel shortage at the steelworks, which was at times quite severe, had partly to be met by the use of wood. Considerable effort was devoted to the development of new firing devices suitable for burning wood. The technical problems were soon mastered, and the shortage of coal did not cause any noticeable decrease in production. However, wood-firing was a heavy economic burden, because wood occupies, per thermal unit, a volume 10–15 times as large as that of coal, and the implication of this in regard to transportation both inside and outside the works is obvious.

The lack of fossil fuel within the country is partly compensated by a relatively good supply of water power. The production in 1952 of electrical energy from hydro-electric power stations amounted to about 20 milliard kWh., and it is estimated that the exploitation of all waterfalls in Sweden would give an annual energy output nearly four times as large.

A comprehensive analysis of the importance of electrical power for the energy requirements of the Swedish steel industry was recently presented by Améen and Lanner.[19] Acording to their work, about one-third of the total amount of energy now consumed by the Swedish iron and steel industry is hydro-electric power, the remainder being obtained from fuels as already described. The Swedish iron and steel industry is thus characterized by a high degree of electrification, and in this respect it differs considerably from that of most other countries. Electrical processes are used in Sweden, both for the production of iron from ore and for steelmaking, to a relatively higher extent than in the large industrial countries. In the following, examples are given to show how the relatively good supply of electrical power and the pronounced scarcity of fuel have directed the technical development into paths peculiar to Sweden. A few data are given here to show the change in the consumption of fuel and power in the last decades. The figures given in Table III, which are taken from Améen's analysis, refer to the total consumption of energy required for the production of 1 ton of finished steel in the years 1923 and 1950. The fuel consumption was 40% lower in 1950 than in 1923. In the meantime, part of the fuel had been replaced by electrical power and the total energy consumption had decreased by 16%.

*The quantities consumed have been recalculated as coal before computing the relative distribution.

Table III

ENERGY CONSUMPTION FOR PRODUCTION OF 1 METRIC TON OF FINISHED STEEL

Year	Power Consumption, kWh.	Fuel Calculated as Coal, kg.	Total Energy Consumption Calculated as Coal, kg.
1923	1010	1920	2320
1952	1970	1170	1950

PRODUCTION OF PIG IRON

The most interesting feature of the Swedish iron and steel industry from a technical viewpoint is perhaps that several different methods are used for the reduction of iron from the ore, and that the coke blast-furnace process is thus not the only one prevailing in this field. The quantitative development of the different main processes used in the twentieth century is shown in Fig. 6, which also gives a curve for the total production of pig iron and sponge iron. The high production at the beginning of the century, which culminated in 1917 at 840,000 tons, was not exceeded until 1944. Since then the production has increased and now amounts to 1·1 million tons.

Charcoal Blast-Furnaces

During most of the period covered by Fig. 6, pig-iron production in charcoal blast-furnaces was the most important method of reduction. Charcoal is an excellent reducing agent as it contains only small amounts of sulphur, phosphorus, and ashes. The charcoal pig iron, which generally contains less than 0·015% S and 0·025% P, is an ideal raw material for the making of quality steels.

to about 2900 million metric tons, containing about 1750 million tons of iron (*see* Table I). Most of this ore quantity consists of lump ore rich in phosphorus and with an iron content of 60% or more. The Lapland deposits are considerably larger than those of Central Sweden.

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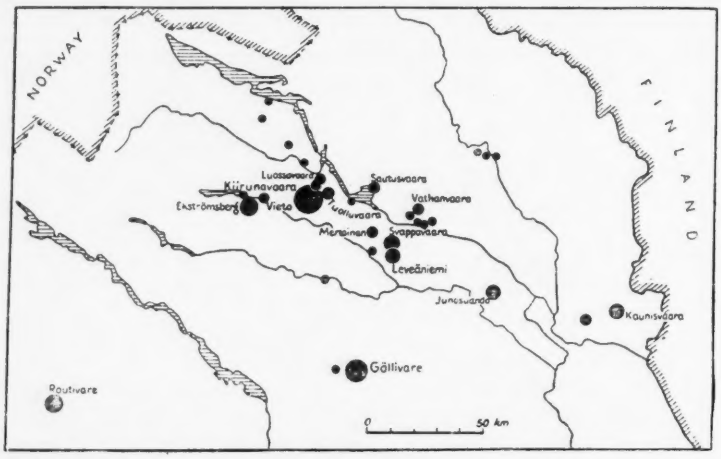

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P_2O_5	0·038	0·020	0·018	0·014	0·009	0·032	0·006	4·48	0·011
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oil, and coal. Blast-furnace coke is produced from coal only to a limited extent.

Statistical data on the consumption of different types of fuel are available over a fairly long period.[18] Figure 5 shows the relative importance of the different fuels used in Swedish iron and steel works during the years 1930 to 1952.* At the beginning of the century charcoal was still the only reducing agent used in the making of pig iron, and was then the most important fuel. Since then it has gradually become of less importance, and in 1952 only about 10% of the total fuel consumption consisted of charcoal. The importance of coke has increased rapidly in later years : half of the total fuel quantity consumed in 1952 was coke and coke breeze. As in the steel industries of the large industrial countries, the use of fuel oil has increased considerably in later years whereas the use of coal, relatively speaking, has decreased. The open-hearth furnaces have to a large extent been rebuilt for burning heavy fuel oils. This is also the case with the reheating furnaces in rolling mills and forging shops. In 1952 no less than one-quarter of the fuel consumption was of oil.

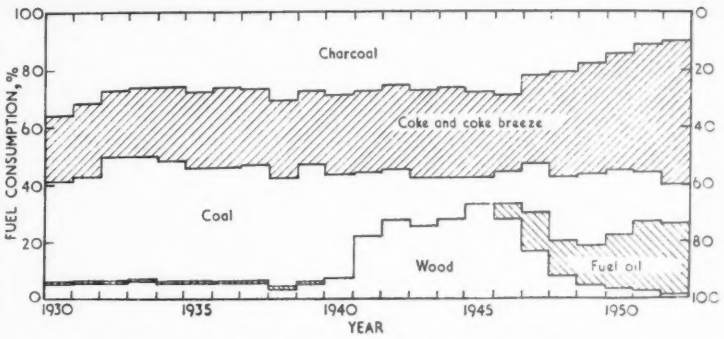

Fig. 5—Consumption of various types of fuel in the Swedish iron and steel industry (relative figures) in the period 1930–1952

During the last war, coal could not be imported to the same extent as it had been earlier. The fuel shortage at the steelworks, which was at times quite severe, had partly to be met by the use of wood. Considerable effort was devoted to the development of new firing devices suitable for burning wood. The technical problems were soon mastered, and the shortage of coal did not cause any noticeable decrease in production. However, wood-firing was a heavy economic burden, because wood occupies, per thermal unit, a volume 10–15 times as large as that of coal, and the implication of this in regard to transportation both inside and outside the works is obvious.

The lack of fossil fuel within the country is partly compensated by a relatively good supply of water power. The production in 1952 of electrical energy from hydro-electric power stations amounted to about 20 milliard kWh., and it is estimated that the exploitation of all waterfalls in Sweden would give an annual energy output nearly four times as large.

A comprehensive analysis of the importance of electrical power for the energy requirements of the Swedish steel industry was recently presented by Améen and Lanner.[19] Acording to their work, about one-third of the total amount of energy now consumed by the Swedish iron and steel industry is hydro-electric power, the remainder being obtained from fuels as already described. The Swedish iron and steel industry is thus characterized by a high degree of electrification, and in this respect it differs considerably from that of most other countries. Electrical processes are used in Sweden, both for the production of iron from ore and for steelmaking, to a relatively higher extent than in the large industrial countries. In the following, examples are given to show how the relatively good supply of electrical power and the pronounced scarcity of fuel have directed the technical development into paths peculiar to Sweden. A few data are given here to show the change in the consumption of fuel and power in the last decades. The figures given in Table III, which are taken from Améen's analysis, refer to the total consumption of energy required for the production of 1 ton of finished steel in the years 1923 and 1950. The fuel consumption was 40% lower in 1950 than in 1923. In the meantime, part of the fuel had been replaced by electrical power and the total energy consumption had decreased by 16%.

*The quantities consumed have been recalculated as coal before computing the relative distribution.

Table III

ENERGY CONSUMPTION FOR PRODUCTION OF 1 METRIC TON OF FINISHED STEEL

Year	Power Consumption, kWh.	Fuel Calculated as Coal, kg.	Total Energy Consumption Calculated as Coal, kg.
1923	1010	1920	2320
1952	1970	1170	1950

PRODUCTION OF PIG IRON

The most interesting feature of the Swedish iron and steel industry from a technical viewpoint is perhaps that several different methods are used for the reduction of iron from the ore, and that the coke blast-furnace process is thus not the only one prevailing in this field. The quantitative development of the different main processes used in the twentieth century is shown in Fig. 6, which also gives a curve for the total production of pig iron and sponge iron. The high production at the beginning of the century, which culminated in 1917 at 840,000 tons, was not exceeded until 1944. Since then the production has increased and now amounts to 1·1 million tons.

Charcoal Blast-Furnaces

During most of the period covered by Fig. 6, pig-iron production in charcoal blast-furnaces was the most important method of reduction. Charcoal is an excellent reducing agent as it contains only small amounts of sulphur, phosphorus, and ashes. The charcoal pig iron, which generally contains less than 0·015% S and 0·025% P, is an ideal raw material for the making of quality steels.

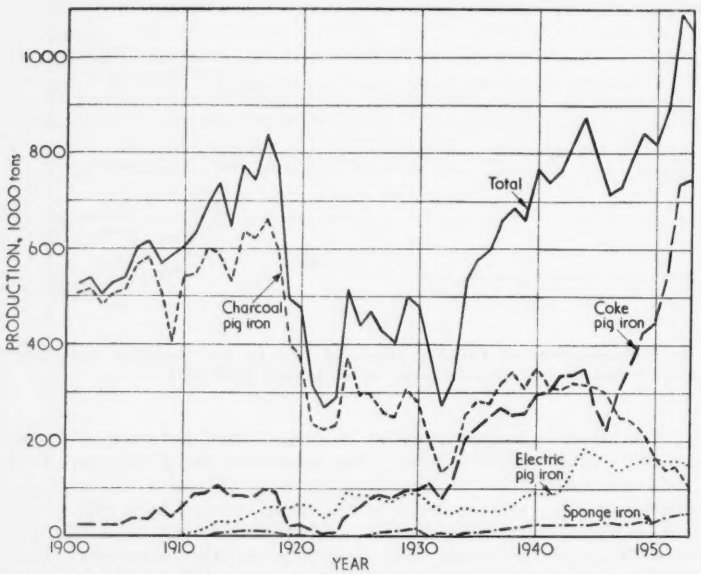

Fig. 6—Sweden's production of pig iron and sponge iron in the period 1900–1953

Comprehensive reviews of technical blast-furnace problems have been published from time to time.[20–22] The charcoal blast-furnaces are comparatively small production units. In 1947[22] the average figure for the hearth diameter was 2·3 m., for the total height 16·7 m., and for the working volume 81 cu. m. The average production was 40 tons per furnace per day.

Great progress has been made in reducing the charcoal consumption. The first sintering plant, according to Greenawalt's system, was built in 1915. Sweden has been a pioneer in the use of sinter in the blast-furnace burden ; as long as 25 years ago all lump ore was replaced by sinter at Hofors,[23] with the result that the charcoal consumption fell considerably. For many years now it has been common practice to use burdens consisting mainly of sinter. Effective concentration of the ores has made possible the introduction of richer burdens, which has led to a considerably reduced slag volume—about 250 kg. per ton of pig iron, and even less in certain cases. Among other measures adopted to reduce charcoal consumption is the addition, before sintering, of the necessary limestone to the mixture, and the use of increased blast temperature by installation of more effective recuperators.[24] The charcoal consumption per ton of pig iron, a figure of decisive importance to the economy of the process, amounted to 850 kg. on an average in 1913. In 1947 the figure had been reduced to 630 kg.

The raw material for charcoal-making is slender logs of coniferous trees. Up to a few years ago such logs could not be used for purposes other than the making of charcoal. By the adoption of new methods, however, it has been possible to utilize this type of timber for the manufacture of wood pulp, thus causing an increase in the price of wood. Also, charcoal-making requires much manual labour, and increased labour costs in recent years have raised the charcoal price. It is now so expensive that in the last few years the charcoal blast-furnace has lost ground in competition with processes using the cheaper coke. At present the charcoal blast-furnace process accounts for only about 10% of the total production of pig iron and sponge iron.

Coke Blast-Furnaces

Coke was first used as a reducing agent in Swedish blast-furnaces at the end of the nineteenth century—at first mixed with charcoal and later alone. For a considerable time the quantity of coke pig iron produced was only moderate, but after the economic depression in the 1930's the production began to increase considerably. The years following the last war show a very marked rise from about 220,000 tons in 1946 to about 750,000 tons in 1953. This increase was due mainly to the marked increase in the production of commercial steels. About 70% of the total production of pig iron and sponge iron now consists of coke pig iron.

The technical development of the coke blast-furnace has been directed mainly towards lowering the fuel consumption. The Domnarfvet Steel Works have been leaders in this field.[25] Good results were obtained principally by preliminary treatment of the ore mixture by sintering. Even 20 years ago lump ore in the burden was entirely replaced by sinter. Some years later the method was introduced of mixing the limestone and other necessary additions with the concentrate before sintering, and nowadays the burden consists of sinter and coke only. For a low coke consumption in the blast-furnace to be obtained it is important that a sinter with a high degree of oxidation is used, *i.e.*, one with a high content of Fe_2O_3. How sinter of this type should be made has been described by Hessle;[26] it is important that the raw material is fairly fine grained, that the fuel in the mixture is finely ground, and that a small amount of fuel is used. If the burden contains only sinter of good quality, the risk of scaffolding is minimized and the temperature of the blast may be kept high. At the recent Symposium on Sinter arranged by The Iron and Steel Institute, Danielsson[27] reported that the coke consumption at Domnarfvet, when producing basic Bessemer pig iron, has now been reduced to an average of 610 kg. (1355 lb.) per ton of pig iron, the temperature of the blast being 895° C. in the ring main. Occasionally the coke consumption has been as low as 580 kg./ton. Very good results have also been obtained at other works. The largest blast-furnace in the country, blown-in about three years ago at Norrbottens Järnverk, Luleå, after introducing a 100% sinter burden, now operates on a coke consumption of 625 kg./ton when smelting basic Bessemer pig iron.

In addition to the saving of fuel, the use of sinter also has the important advantage of leading to increased production. As an example, it may be mentioned that the latest furnace at Domnarfvet has

a production per unit of working volume almost twice that obtained in American blast-furnaces.[27] In retrospect it could perhaps be said that because of the character of the ores the adoption of sinter burdens in Sweden was a rather obvious measure. Sinter burdens have for some time been used also at Scunthorpe in England, where, despite less favourable conditions, very good results have been achieved in regard to both production capacity and fuel consumption.[28]

Most of the Swedish coke pig iron is consumed in the production of commercial steels. At some works, where the coke pig iron is used as raw material also for quality steels, the pig iron is desulphurized outside the blast-furnace. According to an interesting method developed by Kalling,[29] the molten pig iron is treated with finely ground powder of burnt lime in a rotating closed container. An effective desulphurization with sulphur contents of 0·010% or less is obtained.[30] This process is probably unique in the metallurgy of iron, for the molten metal reacts with a substance in the solid state, and no liquid slag is formed.

Electric Pig-Iron Furnaces

The first method of producing pig iron electrically was developed in Sweden. In 1910 Jernkontoret built a pilot-plant furnace of the 'Elektrometall' type[31] and during the next ten years several furnaces of this type were erected at different works.[32] The upper part of the furnace is similar to that of a blast-furnace, and the lower part consists of a wide melting chamber with six or eight electrodes. Charcoal is used as a reducing agent but, as the heat necessary for the process is supplied as electrical energy, the charcoal consumption is only half that of the charcoal blast-furnace. This type of furnace, which may be called the electric high-shaft furnace, cannot work with coke alone as a reducing agent. Therefore only a few units are now in use, and probably no new ones will be built.

The electric low-shaft furnace of the Tysland-Hole type, which was developed in Norway, is used at some works in Sweden. A diagram of the furnace is shown in Fig. 7. A mixture of coke and coke breeze may with advantage be used as a reducing agent, and this furnace is therefore more economical than the high-shaft furnace. The largest units built in Sweden have a power input of 10,000 kW. and an annual production of about 30,000 tons of pig iron.

The production of electric pig iron at present amounts to about 150,000 tons annually. The reason why the electric processes have not become more important is their high power consumption (2000–2400 kWh. per ton of pig iron), which means that they require access to very cheap power. In this respect these processes can hardly compete with the electric sponge-iron method.

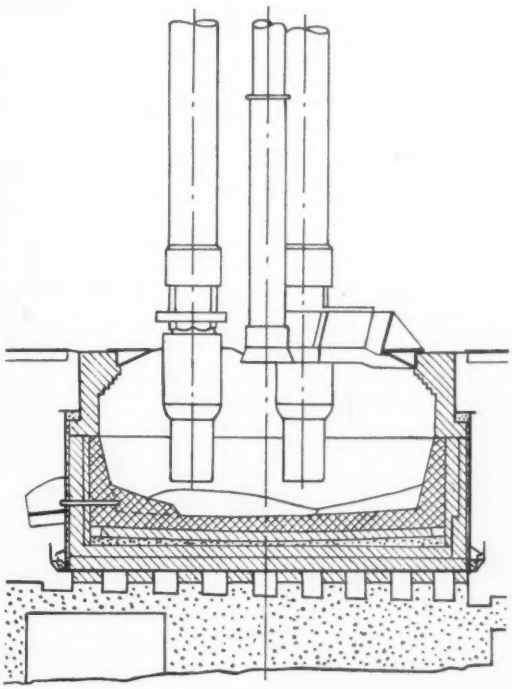

Fig. 7—Tysland-Hole electric pig-iron furnace

PRODUCTION OF SPONGE IRON

Great efforts have been made in different countries to solve the problem of sponge-iron production, *i.e.*, to reduce iron ore on a commercial scale without smelting. It is not by chance that the problem was solved in Sweden, where the scarcity of fossil fuel has directed development towards fuel-saving processes, and where pure ores are available for concentration to a very low gangue content.

The first sponge-iron process was developed by Sieurin, and has been used at Höganäs in South Sweden since 1911. Rich concentrate is reduced in ceramic muffles with coal and coke breeze as reducing agents, and the carbon monoxide formed is combusted outside the muffles, thus generating the heat needed. The sponge iron made at Höganäs has been used partly for the making of quality steels and partly for the preparation of iron powder. A new plant for sponge-iron production, using this method, is now under construction at Oxelösund.

The principle of another sponge-iron method was given by Wiberg[33] in 1918. The reduction is carried out in a shaft furnace with a circulating gas mixture consisting of carbon monoxide and hydrogen (*see* Fig. 8). The gas mixture, at a temperature of 950° C., is introduced into the lower part of the shaft where it meets the descending burden. From the level of the shaft where the burden has been reduced to FeO most of the gas leaves the furnace and is led to a carburettor. This is an electrically heated container, filled with charcoal or coke, where the carbon dioxide and water vapour in the gas are reduced to carbon monoxide and hydrogen. The circulation of gas through the furnace shaft and the carburettor is brought about by a blowing fan, with a working temperature of 700–800° C. On its way from the carburettor to the furnace shaft the gas passes through a container of dolomite, where the sulphur content of the gas is absorbed. A small amount of gas is led through the upper part of the

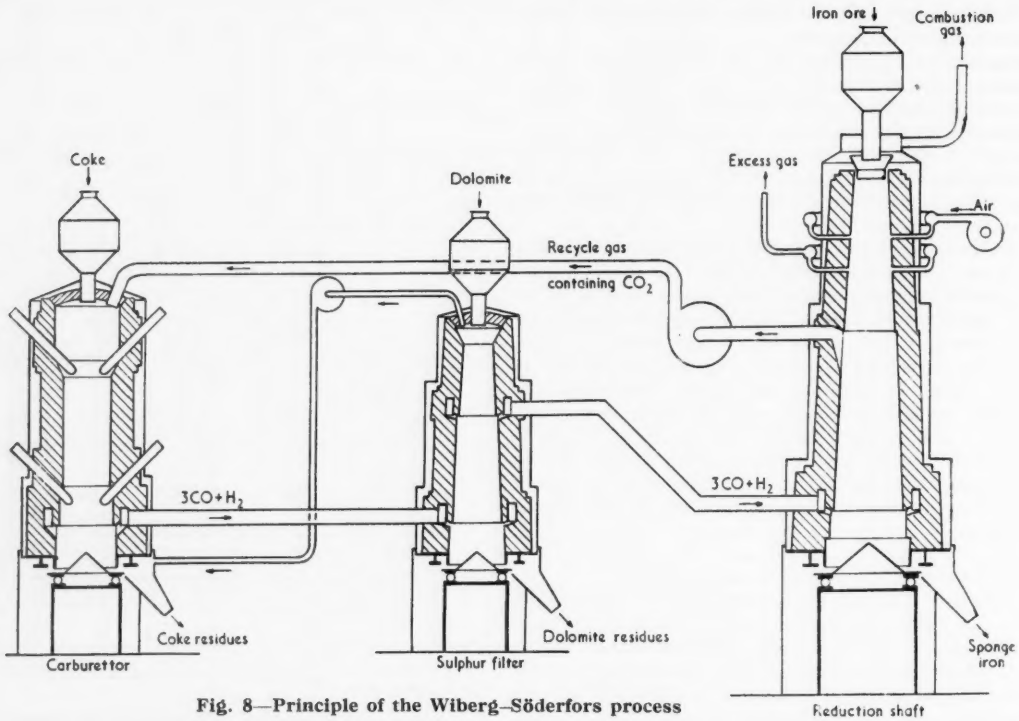

Fig. 8—Principle of the Wiberg–Söderfors process

furnace shaft in which the burden is reduced to the FeO stage. In the top part of the shaft the burden is preheated by combustion of the gas. The sponge iron produced is discharged at the bottom of the shaft, filled into closed containers, and transported to the steel furnaces or left to cool down.

After the method had been tried on a pilot-plant scale in the 1920's, the first full-size furnace was built at Söderfors in 1932. The carburettor and the blowing fan caused great problems of design which, however, were solved by concentrated effort during the following years. In 1943 Améen[34] reported that the difficulties had been overcome and that the furnace had been working continuously for some time with charcoal as reducing agent in the carburettor. The process has since been still further developed, especially by Stålhed.[35] The reducing agent is now blast-furnace coke, and the coke consumption is about one-third of that in the coke blast-furnace (*see* Table IV).

The demands on the strength of the burden during reduction are higher in the Wiberg method than in blast-furnace practice. In a few cases there has been a tendency for pan sinter made from certain ores to choke the shaft in the reduction zone. The problem was solved in a novel way after pelletizing was introduced in Sweden on the initiative of Tigerschiöld.[36] Sintered pellets prepared in a certain way were found to be more suitable than ordinary sinter, and will be used in all Wiberg furnaces.

The gangue of the ore is not removed in reduction, and the gangue content should therefore be kept low. The raw materials used are rich magnetite concentrates with up to 70% Fe (*cf.* Table II). The sponge iron obtained generally has an iron content of about 90% or rather more. About 90% of the iron is present in metallic form, and the rest as FeO. As the product does not absorb any impurities from the fuel during reduction, extremely low contents of phosphorus and sulphur are obtained, of the order of 0·005% of each. Sponge iron is a very good raw material for the production of quality steels, and can with advantage be used together with pig iron or scrap in open-hearth or electric steel furnaces.[37]

Table IV shows fuel and power consumption figures for different methods of ironmaking. These figures do not, of course, provide a sufficient foundation for an economic comparison of the different processes, but they may serve to explain why the interest in sponge iron is so large in Sweden. Sponge iron will to a large extent become the substitute for charcoal pig iron in the production of the quality steels.

Table IV

APPROXIMATE FUEL AND POWER CONSUMPTION PER TON OF PIG IRON AND SPONGE IRON IN DIFFERENT REDUCTION PROCESSES

Process	Charcoal, kg.	Blast-Furnace Coke, kg.	Coke Breeze, kg.	Power Consumption, kWh.
Charcoal pig iron	630			80
Coke Pig Iron		650		110
Electric pig iron, high-shaft furnace	310			1900
Electric pig iron, low-shaft furnace		200	200	2300
Sponge iron, Wiberg–Söderfors		230		1050
Sponge iron, Höganäs			650	

The total production of sponge iron during 1953 was about 50,000 tons. For 1954 the production capacity is estimated at almost 200,000 tons, as a total of seven new sponge-iron plants will probably be in operation.

STEELMAKING

Compared with other countries the steelmaking of Sweden, like its ironmaking, shows characteristic features in regard to the relative significance, from a quantitative point of view, of the various processes employed ; the difference from other countries, however, is more one of degree than of kind, and is caused mainly by the fuel situation.

To a larger extent than is the case in most other countries, the steel industry in Sweden is directed towards the production of quality steels. The reason for this is historical, and dates from the time when the low phosphorus and sulphur contents of the raw materials gave a unique position to the country. This tradition is still alive in the minds of steelmakers at all levels as a deeply rooted understanding of the importance of quality. Skill and technical knowledge mean more now than in the old days when the raw materials gave a privileged position to the country.

The quantitative development during the twentieth century of the Swedish production of wrought iron, steel ingots, and castings is shown in Fig. 9. The total annual production, which during the First World War reached a peak figure of 750,000 tons, then declined very rapidly to a low point at 250,000 tons in 1921. Since that time there has been a rising tendency on the whole, except during the depression years in the 1930's when a temporary decline occurred, and during the last war when the production was maintained at a fairly constant level. Since 1947, when 1·2 million tons were made, the total production has increased by about 50% to nearly 1·8 million tons in 1953.

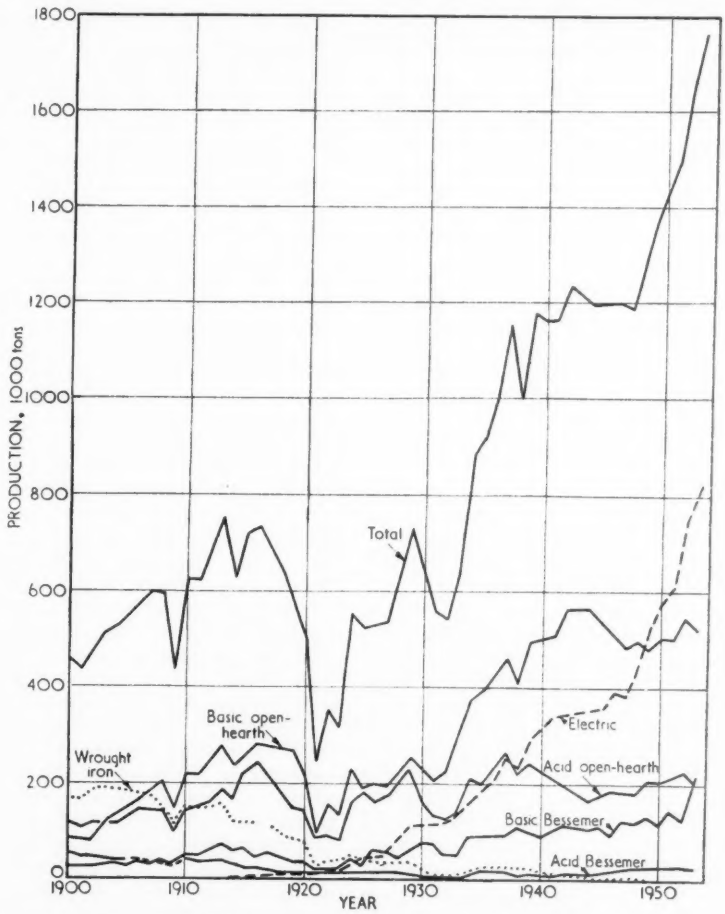

Fig. 9—Sweden's production of wrought iron, steel ingots, and castings in the period 1900–1953

Wrought Iron

Wrought iron was still made in considerable quantities during the first part of the century. It is worth mentioning that only in 1908 was the wrought-iron production surpassed in quantity by one of the ingot steel methods. Several thousand tons of wrought iron are still being produced annually.

Acid Bessemer Steel

As mentioned earlier, the acid Bessemer process was introduced in Sweden by G. F. Göransson, who made the first satisfactory Bessemer heat as early as 1858. The production of Bessemer steel reached its maximum in 1890, when it amounted to 95,000 tons. The process then became less and less important until a revival was experienced with the erection at Fagersta in 1946 of a new plant with two 20-ton converters. At this plant the converter is kept in a tilted position during the blow, with ore addition in the converter, giving a steel with a low nitrogen content (about 0·008%).[38]

The classical Swedish acid Bessemer process was based on charcoal pig iron with a high manganese content (about 3%) and low phosphorus and sulphur contents. Another difference compared with foreign practice was that the blowing was interrupted when the required carbon content in the steel had been reached ; carburizing in the ladle was not used, nor was addition of manganese alloys.[39] The steel, which was exported mainly in ingot form, was used as tool steel and was characterized by good forgeability and good hardening properties. The production of tool steel according to this process is now of less importance. The few existing Bessemer plants are largely used in the making of duplex metal for open-hearth or electric steel furnaces.

Basic Bessemer Steel

For a number of years the basic Bessemer process has been in use at one Swedish works only, the Domnarfvet Steel Works. There it has been employed partly for direct ingot production and partly for duplex melting with electric steel furnaces. In connection with the recent comprehensive expansion and modernization of these works, a new basic Bessemer plant has been erected with four 28-ton converters.

As in some of the foreign basic Bessemer plants during later years, considerable work at Domnarfvet has been devoted to the development of the basic Bessemer process—in the first place aiming at improving the quality of the steel. Reports on the results obtained have been published by Kalling and his co-workers.[40, 41] Blowing with oxygen-enriched air combined with the addition of ore gives a reduced nitrogen content in the steel of less than 0·008%. A new large oxygen plant is now operating, and the use of oxygen is normal practice ; it is economically justified solely by the fact that the process may be conducted with a larger addition of scrap or iron ore than in air blowing. If a nitrogen-free mixture of oxygen and carbon dioxide is used during the last blowing period, the nitrogen content can be still further lowered. Interesting experiments have also been made to refine pig iron with pure oxygen in a rotary furnace. By this method, very effective dephosphorization is obtained and also an extremely low nitrogen content in the steel; moreover, the smoke formation is not so disturbing.

In 1953 another basic Bessemer plant was put in operation at the State-owned Norrbottens Järnverk at Luleå. When both these plants reach full capacity the annual production of basic Bessemer metal will be of the order of 750,000 tons.[42] A considerable part of this quantity will be used as liquid charge in electric steel furnaces.

Acid Open-Hearth Steel

In the last decade the production of A.O.H. steel has been maintained at a fairly constant level of about 200,000 tons annually. The proportion of this process of the total steel ingot production is as high as 11%.

The process still occupies a strong position in the production of quality steels, because of the excellence of A.O.H. steel for certain purposes, *e.g.*, for machine parts subjected to high stress and for hardened products in strip or wire form. Thus, ball bearings made from A.O.H. steel have a considerably longer life than those made from electric steel.[43]

The raw material for the process consists of pig iron, sponge iron, and O.H. scrap. A high percentage of pig-iron in the charge is characteristic. The Swedish method of working the heat differs considerably from that used abroad. The lining consists of pure quartz with low amounts of impurities, and therefore has good refractoriness at high temperatures. During the refining period the temperature of the bath is allowed to rise so much that the carbon dissolved in the steel reduces the silica of the lining, thus causing a rise in the silicon content of the bath. This procedure, called the ' silicon boil,' is controlled by estimating the silicon content from spoon samples. Part of the silicon reduced is oxidized at the contact surface between steel and slag, and consequently the silica content of the slag rises and reaches the saturation point. The slag composition becomes almost the same from heat to heat, and the variation of steel composition and of steel temperature is small. Additions of limestone or fluorspar are not made. Nowadays, oxygen is sometimes used to shorten the time from ' clear melt ' to the beginning of the boil.

Interest in the metallurgy of the A.O.H. process was considerable in Sweden in the 1930's, following the publication of the results of fundamental laboratory experiments by Körber and Oelsen.[44] Carbon refining was studied by Kalling and Rydberg,[45] who elucidated the rôle of carbon and oxygen in the process and the mechanism of the silicon boil. Comparative investigations on the behaviour of hydrogen and nitrogen in different steelmaking processes were also carried out.[46, 48]

Formerly, A.O.H. furnaces were generally built for a capacity of 20 tons, whilst more recently larger furnaces have also been brought into use. Oil firing is usually employed. The instrumentation has been enlarged and improved.

Basic Open-Hearth Steel

The annual production of B.O.H. steel increased rapidly from about 100,000 tons in 1921 to 500,000 tons in 1939. Since then the output has been fairly constant. About 30% of Swedish steel is now made in B.O.H. furnaces.

The process is used mainly for making commercial steels, the charge consisting largely of commercial scrap. The furnaces are usually oil-fired, and the walls and roof of the furnace chamber are built of basic

refractories. A fairly complete instrumentation is generally part of the furnace equipment. Modern furnaces often have a capacity of 50–60 tons, and at Fagersta an 80-ton furnace has been in operation for some years. When a cold charge is used, the output of a 55-ton furnace is about 7 tons of ingots per hr.

Part of the steel manufactured is rimming steel. Of great importance to this type of production were the investigations made by Hultgren and Phragmén[49] in the 1930's. Their theory of the solidification of rimming steel was later confirmed by English investigators, notably by Swinden and his co-workers.[50–52] Lately there has been a decline in rimming steel production in favour of killed and semi-killed steel.

Electric Steel

The good supply of water power in Sweden accounts for the fact that the price of electric power is comparatively low in proportion to the price of imported fuels. The relatively low price of hydro-electric power has in turn given the electric steel processes an unusually strong position. Since 1920 the production of electric steel has shown an almost unbroken increase, lately so intense that the output has more than doubled over the last six years. In 1953 the production of electric steel ingots amounted to about 800,000 tons—46% of the total ingot production.

Electric arc furnaces are used to a considerable extent in the making of commercial steels ; the charge may consist either of commercial scrap and pig iron or of liquid pre-blown metal from acid or basic Bessemer furnaces. The heat is finished under an oxidizing slag, and the working of the heat resembles that applied in the B.O.H. furnace.

For quality steelmaking—the ' classical ' field of the electric arc furnace—this type of furnace is much used in Sweden, and all its varied metallurgical possibilities are being utilized in this connection. Since the war the use of oxygen for refining has been introduced at several works. An innovation which is particularly important for quality steelmaking is the electrodynamic stirrer developed by Dreyfus[53] at Surahammar. The stirrer, working as a segment of the stator of a big electric motor, is placed under the non-magnetic bottom plate of the furnace and causes a mild stirring of the steel bath (*see* Fig. 10). Temperature and concentration gradients formed in the bath during the reducing period are rapidly equalized by the stirring, and the reactions taking place at the contact surface between steel bath and slag are accelerated.[54, 55] The reducing period can be shortened accordingly. Electrodynamic stirring has been in use for some years in several Swedish electric arc furnaces, with good results, and favourable experience is reported also from a 90-ton American furnace, where the method is now being applied.[56]

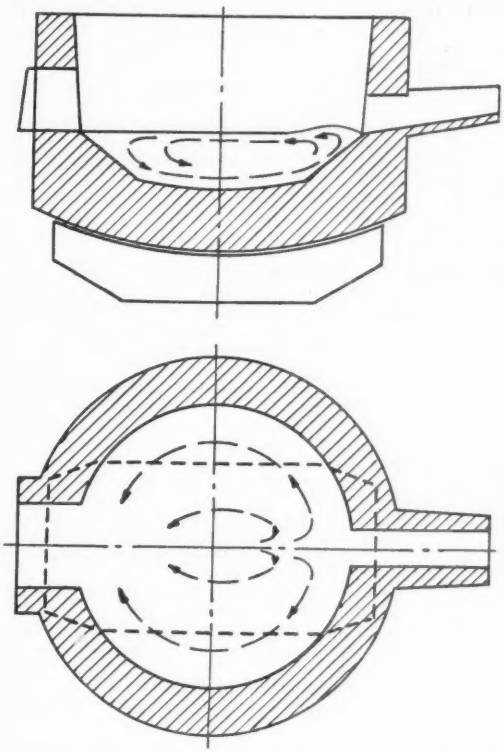

Fig. 10—Motion produced in bath by electrodynamic stirrer.

Basket charging is used at most Swedish arc furnaces. The furnace size varies according to the use, from very small units to capacities of 30–40 tons. A tendency to increase the transformer capacity and the secondary voltage is becoming more and more noticeable.

The H.F. induction furnace is relatively more used in Sweden than in other countries ; about 5–6% of the total ingot production originates from this type of furnace. It is of special advantage in the making of high-alloy steels, where the saving of alloying elements is an important factor. The H.F. induction furnaces at the steelworks usually have capacities of 5–8 tons. The largest unit is at Bofors, where two furnace bodies, each of 12-ton capacity, have been in operation for some time. These furnaces are probably the biggest of their kind in the world. The development in the field of H.F. melting in Sweden has been led by the firm ASEA, which holds a prominent position as furnace manufacturers.

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Aspects on Pelletizing of Iron Ore Concentrates

By Magnus Tigerschiöld

SYNOPSIS

A brief survey is made of the general development of the method of agglomeration of iron ore concentrates in Sweden, and a report is given of the theoretical work done in Sweden and in other countries in regard to the factors affecting the performance of the pelletizing method. The technical development of the shaft-furnace process is also reviewed, and future uses of the method are suggested. There is still much work to be done in laboratories, as well as on the design and operation of full-scale furnaces, but it seems clear that pelletizing in shaft furnaces will become an important complement to existing methods of agglomeration. This will be the case especially when utilizing rich magnetite concentrates. For treating hematite concentrates, pyrite residues, and not too rich magnetite concentrates that tend to stick, the use of sintering machines for baking the pellets might be a solution. 935

Introduction

At the Symposium on Sinter arranged by The Iron and Steel Institute in November, 1953, important contributions were made in regard to the sintering of iron ores by classical methods and the use of a high percentage of sinter in coke blast-furnaces. For the first time it was convincingly made clear to an international assembly that the use of 100% sinter is a very efficient way of increasing the production and decreasing the fuel consumption in blast-furnaces. At this Symposium, reference was made to Swedish experience in this field.

Review of Briquetting and Sintering in Sweden

At the end of the last century Dr. Gustaf Gröndal, a Swedish engineer, invented the well-known briquetting method. The first tunnel kilns were built at Pitkaränta in Finland (in 1900) and at Bredsjö in Sweden (in 1902). The method was rapidly exploited, and by 1915 about 350,000 tons of concentrates per year were briquetted in Sweden and kilns had been erected in many foreign countries. At that time, however, the more economical sintering methods were introduced in Sweden, and nearly all the briquetting furnaces have now been replaced by sintering plants.

An 80% sinter burden was used regularly as early as 1921 by Th. Quennerstedt in a new charcoal blast-furnace at Söderfors, where 100% sinter was also tried. A description of the blast-furnace plant at Söderfors was published in 1929 by Clements in his textbook on blast-furnace practice.[1] In 1932 the furnace was pulled down to give place to the first full-scale Wiberg sponge-iron furnace, and in 1953 the Greenawalt plant was replaced by a pelletizing furnace.

In 1929 Cedervall[2] started experiments using 100% sinter in the charcoal blast-furnace at the S.K.F. plant at Hofors. He made his sinter from magnetite concentrates of the right iron content, and he developed his sintering plant to a high degree of efficiency. The work done by Cedervall was very soon recognized as a most important contribution to the Swedish iron and steel industry. Nearly all the existing sintering plants in Sweden were improved, extended, or rebuilt for higher production ; also, new plants were erected. By 1936 the average proportion of sinter used in Swedish blast-furnaces approached 80%, and it is now 90% of the ore burden.

Great savings were made in this way, owing to the fact that well-oxidized light-burned sinter has a much better reducibility than magnetite ores in lump form and also than a hard-burned sinter, as ferric oxide is more easily reducible than magnetite. This fact has long been realized in Sweden. Every Swedish blast-furnace used to have special roasting furnaces where the lump ore—mainly dense magnetites—was roasted before being charged into the blast-furnace. The roasting process was controlled in such a way that a high degree of oxidation was achieved. (The Swedish term ' oxidationsgrad ' (degree of oxidation) was defined as early as 1874 by Tholander[3] as the amount of oxygen actually combined with Fe^{III} and Fe^{II} divided by the amount of iron-bound oxygen that the ore would contain if the whole of the iron occurred as Fe^{III} (Fe_2O_3). On the basis of this definition the following simple expression can be deduced :

$$\text{Degree of oxidation} = \left(1 - \frac{Fe^{II}}{3\,Fe_{tot}}\right) \times 100\,\%.$$

Thus, the degree of oxidation is 66·6% for pure FeO, 88·9% for Fe_3O_4, and 100% for Fe_2O_3.)

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In 1946 Hessle[4] presented a remarkable paper on sintering in which he emphasized the importance of using a highly oxidized sinter and described and discussed the methods of its manufacture. Hessle's research work was limited to the manufacture of sinter for charcoal blast-furnaces. Results achieved in the coke blast-furnaces at Domnarfvet were published in 1946[5] and 1949[6] in leading English and American journals, but it was some time before the possibility of applying the Swedish practice in foreign countries was fully recognized. The opinion that sinter must be extremely hard-burned to be strong enough for blast-furnace use has been too strongly emphasized. In 1929, for instance, Schwartz[7] wrote :" In all satisfactory sinter the amount of hematite is small, perhaps not over 2 or 3%," whilst Swedish sinter today contains more than 90% of hematite.

The trials made by Elliot *et al.*[8] at Appleby Frodingham, the results reported by Danielsson[9] from the Domnarfvet coke blast-furnaces, and the scientifically convincing explanation by Edström[10] of the mechanism of reduction will probably attract worldwide interest in the better preparation of burden for blast-furnaces, and will thus lead to possibilities of great savings through lower coke consumption and higher production.

The clever device of mixing finely crushed limestone with the ore before sintering was tried abroad at an early stage, but its significance was first recognized in Sweden, where it is now common practice. Much work, scientific as well as technical, has still to be done in this field, and probably there will be many improvements in methods of ore beneficiation and of preparing the blast-furnace burden.

This introduction serves to give a background and an explanation of the great interest with which Swedish ironmakers have welcomed every new development in this field.

DEVELOPMENT OF THE SHAFT-FURNACE PELLETIZING PROCESS

When in 1946 the results of the work done by Davis, Firth,[11] Wade, and others at the Mines Experiment Station at the University of Minnesota became known in Sweden, a committee* was immediately formed by Jernkontoret to investigate the possibility of using the pelletizing process in Sweden. The Brackelsberg method tried at Rheinhausen, Germany, in about 1932 was known, and the reasons why these experiments could not be successful were understood, but the fact that the pelletizing method was originally invented in Sweden by Andersson as early as 1912 had been overlooked. With the exception of his Swedish letters patent,[12] however, none of his results had been published ; they were not very promising, for reasons which are quite obvious to-day.

The members of the committee soon came to the conclusion that the pelletizing process, in the form in which it was tried in the U.S.A., showed great possibilities of being developed into a suitable method for treating fine iron ore concentrates. The simplicity of the method, the good heat economy, and the high degree of oxidation (over 99·0%) of the product were particularly interesting features. The possibility of making a strong product which could stand the handling and transportation from the mine to the consumer was also a great advantage.

The product appeared to be an ideal raw material for sponge-iron shaft-furnaces of the Wiberg type, and at that time the Sandvikens Jernverks AB had decided to build a sponge-iron furnace in which they could use the very rich and finely ground concentrates from the Bodås mine as raw material. Trials had shown that the classical sintering methods could be used, but the results were not quite satisfactory. This was a strong reason for trying the pelletizing method. Together with Jernkontoret, Sandviken accordingly erected a small pilot pelletizing plant ; the more fundamental research work was carried out by Jernkontoret at the laboratories of the Institute of Technology, Stockholm. It took nearly three years of hard work, however, before pellets were produced which were good enough to use in the Wiberg sponge-iron shaft-furnace. The pelletizing method, which at first seemed very simple, was found to be much more intricate than had been expected.

The pelletizing process at its present stage is performed in the following way. Iron ore concentrates milled to a very high degree of fineness and with a water content of about 9% are fed into a cylindrical, nearly horizontal, rotating drum. Small spherical balls are formed in the drum, in the same way that snowballs can be formed. At the discharging end of the drum a sieving device is arranged by means of which undersized balls and concentrate that has not formed balls are removed and brought back to the charging end of the drum while balls of adequate size are fed into a shaft-furnace.

The green balls are not very strong and must be handled carefully in transportation and when feeding into the furnace. The balls are dried in the upper part of the furnace, and it is necessary to carry out this drying quickly and to keep the hot zone in the furnace at a rather high level, as the dried balls are weak and cannot withstand the pressure of a high column of balls. The maximum temperature in the furnace should therefore be kept at a level about 2 ft. below the surface. The fired pellets should be allowed to cool slowly, and the sensible heat is recuperated by using part of the combustion air for cooling the pellets. The furnace is heated by burning oil or gas in combustion chambers on the sides of the furnace.

From this very brief description of the process it is clear that the properties of the green and the dried balls must be kept within narrow ranges and at certain standards, to obtain fired pellets of a good quality. Although the analysis of the concentrate is usually of small significance for the balling operation, it is of great importance for the furnace operation and for the properties of the fired pellets. For the balling operation the water content and the fineness of the concentrates are most important, whilst the size of the balls is significant for the rate of production both of the balling drum and of the furnace. The use of

*Originally the committee was constituted as follows : M. Tigerschiöld (Chairman), B. Hessle, S. Mörtsell, and M. Wiberg. Since then G. Helmer, N. Winblad, P. A. Ilmoni, K. G. Görling, S. Fornander, B. Björkvall, J. Stålhed, L. O. Uhrus, H. Kihlander, J. Uggla, and E. von Hofsten have been appointed or co-opted members.

additives can influence both the properties of green balls and the rate of production and quality of product when burning the pellets.

The Balling Operation

Jernkontoret first studied methods of rolling and the properties of green balls. The first report on their results was presented at the A.I.M.E. meeting at Cincinnati in April, 1950,[13] and was also shortly afterwards published in *Jernkontorets Annaler* in a somewhat improved form. A positive result of this investigation is the theory put forward by Ilmoni[13] that the strength and density of the green balls are due to a large extent to the surface tension of the water and the action of capillary forces. This hypothesis was opposed in the discussion at the meeting at Cincinnati, but after a written reply had been published the theory seems to have been generally accepted.

The concentrate to be used should be milled to a high degree of fineness. In the United States as well as in Sweden the concentrates used for pelletizing are milled to about 80% of −250 mesh.

The water content of the concentrate to be balled must be kept within a narrow range at a value which varies with the true density, the fineness of the concentrate, the grain-size distribution, and the shape of the single grains. The variations in water content should not exceed $\pm 0{\cdot}25\%$. Usually, and especially in the U.S.A., the water content is given as the percentage of water in the moist concentrate, but frequently, as in Tigerschiöld and Ilmoni's paper,[13] it is calculated in relation to the percentage of dry substance. (The relation between the water content on a wet basis and that on a dry basis is

$$W_d = \frac{100W_w}{100 - W_w}\%$$

where W_d is the water content counted on the dry substance and W_w is the water content in the moist concentrate. As an example, if W_w is 10%, W_d is 11·1%.)

For magnetite concentrates the maximum crushing strength of the green balls is attained at a moisture content W_w in the wet concentrate of 6–7%, but as the brittleness of the balls increases with the crushing strength the water content must in practice be kept somewhat higher. In the U.S.A. the water content is usually kept at about 10%, and in Sweden at about 8%. For pyrite residues, where the individual grains have a porous structure, the water content must often be kept at 16–20%. Very fine concentrates are not easy to filter, and it has been a difficult problem to reduce the water content in the concentrates to a low enough level. However, by using a vacuum of 25–26 in. of Hg on the filters, and special nylon cloths, this problem has been satisfactorily solved. In the beneficiation of iron ores wet methods have hitherto been applied for milling as well as for the concentration. It would probably be an advantage in pelletizing if the beneficiation could be made by the dry method, as the water content of the concentrate could then be more accurately controlled.

The dimensions of the drum, the inclination, the peripheral speed, and the thickness of the layer of material kept in the drum are of great significance in regard to the quality of the green balls and the production per unit. According to present experience the length of the drum should be about 2·3–3 times the diameter. The production depends on the required size of the balls. At Bodås, where $1\frac{1}{4}$-in. balls are made and the diameter of the drum is 4 ft., the production is about 120 metric tons per day, whereas in the U.S.A., where $\frac{7}{8}$-in. balls are standardized, the production in 8-ft. drums is about 250 tons per day. The inclination of the drum should be 2–3·5°, and the peripheral speed of the drum should be kept at about 270 ft./min. when $\frac{7}{8}$-in. balls are made. In Sweden the drums are run at peripheral speeds of 70–120 ft./min. for rolling $1\frac{1}{4}$-in. balls. If the speed is too low the mass in the drum slides and no balls are formed. If the speed is too high, cascading takes place and the balls are damaged. The drum should be scraped, keeping a fairly uniform layer of wet concentrate on the inner surface. In the U.S.A. a horizontally back and forward moving toothed knife is generally used, and in Sweden rotating spiral scrapers are used. Usually the sieve is cylindrical and consists of rings or a spiral of the same diameter as the drum ; it can be driven separately or can be attached to the drum as an extension. The amount to be circulated also depends on the required size of the balls and can vary between wide limits. It is remarkable that under certain conditions the circulating quantity can be as high as 400%.

When designing and operating the balling equipment every precaution should be taken not to damage the balls formed. The circulating masses, which contain undersized balls, should also be handled carefully. Even small cracks cause trouble when drying and baking the balls in the shaft-furnace. The influence of ball size and of certain additives has been investigated by Ilmoni and Uggla[14] and will be discussed later.

In all pelletizing plants for iron ore concentrates hitherto constructed, cylindrical drums have been used for the balling operation. In the cement industry, however, a different kind of balling equipment, originating from Czechoslovakia, has found increasing application. The equipment consists of an inclined table with a cylindrical edge ; it is manufactured in Europe by, among others, Loesche at Düsseldorf, Germany, and Ludw. von Roll'schen Eisenwerke at Gerlafingen, Switzerland. A small-scale test with Swedish concentrates gave promising results, and a big table, 12 ft. dia., will shortly be tried in full-scale operation at Malmberget.

The Burning of Pellets

The green balls must be strong enough to withstand transportation and charging into the furnace. They should stand a fall of about 1 m. without cracking and without being noticeably flattened. As seen in Table I, 20-mm. dia. green balls have a crushing strength of about 2 kg. When charged into the furnace the balls should withstand a rapid rise of temperature without cracking. Ilmoni and Uggla[14] have made a thorough investigation of the resistance to rapid drying of green balls, and have found that this property can be strongly influenced by additives.

Table I

INFLUENCE OF ADDITIVES ON PROPERTIES OF 20-mm. PELLETS MADE FROM MALMBERGET CONCENTRATES

Property	No Additive		Bentonite		CaO				$CaCl_2$		NaCl	Borax
	a	b	0·25%	0·5%	0·5%	1·0%	3·0%	5·0%	0·25%	0·5%	0·25%	0·15%
Green Balls												
Water content W_w, %	6·5	6·9	7·1	6·9	6·5	6·5	6·6	6·5	6·6	6·6	6·7	7·0
Apparent density, g./c.c.	3·59	3·52	3·54	3·47	3·53	3·55	3·49	3·37	3·56	3·57	3·55	3·52
Porosity, %	30·3	31·5	31·0	32·1	30·8	29·9	29·4	30·3	30·7	30·2	30·8	31·3
Resistance to dropping, cm.	100	100	100	105	110	115	125	125	100	105	105	90
Resistance to knocking—												
No. of falls from 10 cm.	25	22	30	35	22	29	36	32	17	17	20	25
Crushing strength, kg.	2·2	2·1	2·9	3·5	2·6	2·8	3·2	2·9	2·0	2·0	2·0	2·4
Dry Balls												
Crushing strength, kg.	0·4	0·4	2·9	6·8	3·5	4·5	8·3	8·0	1·4	2·0	1·3	0·7
Resistance to rapid drying—												
shock temperature, °C.	175	175	500	450	n.d.	n.d.	n.d.	350	350	350	+600	+600
Fired Pellets												
Apparent density, g./c.c.	3·97	3·95	n.d.	n.d.	n.d.	n.d.	n.d.	4·0	3·94	3·95	3·92	n.d.
Shrinkage at burning, %	5·1	5·2	6·1	7·3	n.d.	n.d.	n.d.	13·6	5·3	5·4	5·2	5·9
Crushing strength, kg.	1130	1520	1750	1970	1260	1770	1930	2310	1250	1250	1650	1475
Strength at reduction:												
Material +10 mm., %	89	90	83	96	94	99	91	99	94	93	19	20
,, +6 mm., %	89	92	88	96	97	99	95	99	96	94	31	24
Reducibility:												
Time for 50% red., min.	21	n.d.	n.d.	24	21	22	24	26	n.d.	19	20	n.d.
,, ,, 95% red., min.	85	n.d.	n.d.	90	120	95	96	98	n.d.	76	78	n.d.
Change of volume at reduction, %	+60	n.d.	n.d.	+33	−6	+8	−1	+9	n.d.	+18	+110	n.d.

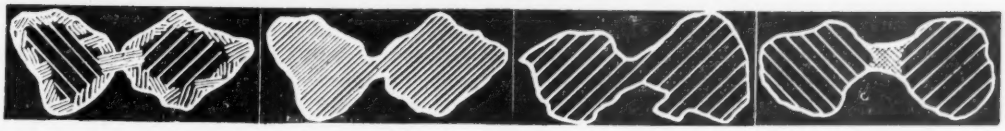
n.d. = not determined

The determination of the resistance to rapid drying is made in the following way. A laboratory furnace with a great heat capacity is heated to a certain temperature. Ten or more green pellets are quickly inserted in the furnace and the behaviour of the pellets is observed. If none of the pellets cracks, the furnace temperature is raised and a new batch of green pellets is put into the furnace. The experiment is repeated until a temperature is reached at which a few pellets show cracks. This temperature is called the shock temperature of green pellets. When no additives are used the shock temperature of Swedish rich concentrates is 175–275° C.; with additives it can be raised to more than 600° C.

When using magnetite concentrates, slight oxidation and cementation of the single grains take place at temperatures as low as 200–300° C. This is one of the most important reasons why magnetite is a more suitable raw material than hematite. Another reason is that the reaction $2Fe_3O_4 + O = 3Fe_2O_3$ is slightly exothermic. In the oxidation of 1 kg. of Fe_3O_4, 111·4 kcal. of heat are evolved. When magnetite pellets made from finely milled concentrates are heated in an oxidizing atmosphere, complete oxidation in the centre of the pellet takes place if the temperature is not raised too quickly. Thus, the temperature in the centre will finally rise to a somewhat higher level than on the surface. If pellets made from hematite concentrates are heated no oxidation takes place, and to achieve a high enough temperature in the centre a higher temperature must be applied on the surface of the pellets, which increases the risk of pellets sticking together. Also, when using magnetite it is easier to maintain a sufficiently high temperature in the centre of the furnace than if the green balls are made from hematite. The heat consumption, which in large furnaces is usually about 120,000–150,000 cal./ton for magnetite, will be nearly twice as high for hematite. An addition of coal is regularly made in full-scale American pelletizing plants operating at a high production rate. For magnetite the additions have been kept within the range 1·0–1·5%. According to Swedish experience, however, in medium-sized furnaces an addition of coal should, if possible, be avoided when treating magnetite concentrates. The combustion of the carbon is difficult to control and the evolution of carbon dioxide might cause cracks in the pellets. Also, when adding coal the oxidation of the magnetite will be delayed or made impossible in the centre of the pellets. This might cause concentric cracks in the pellets and a dense structure, and always results in a lower degree of oxidation ; this in turn will decrease the reducibility of the pellets. If hematite is used as raw material an addition of coal is probably necessary, at least in large furnaces.

It is obvious that if the concentrate contains sulphur an evolution of heat occurs when the pellets are fired, and it has been found that pyrite residues containing about 1% of sulphur are easier to pelletize than those with an appreciably lower content of sulphur.

Cooke and Ban[15] at the University of Minnesota have studied the metallurgy of the pelletizing process and have shown that in fired pellets made from magnetite concentrates four different types of bond can exist (*see* Fig. 1) :



	(i)	(ii)	(iii)	(iv)
Composition of Bond	Hematite	Hematite	Magnetite	Slag
Mode of Formation	Oxidation of hematite	Recrystallization of hematite	Recrystallization and grain growth of magnetite	Formation of iron silicates
Atmosphere	Oxidizing	Oxidizing	Neutral	Neutral or slightly oxidizing
Temperature of Formation, ° C.	> 200	> 1100	> 900	> 1000

Fig. 1—Different bonds between single grains in a pellet made from magnetite concentrate

(i) The bond consists of hematite between partly oxidized grains of magnetite. This binding starts in an oxidizing atmosphere at temperatures as low as 200–300° C. The oxidation first occurs in certain crystallographic directions on the surface of the grains. Small hematite crystals are formed on the surface of the original uniform magnetite crystal. Because of the great mobility of the atoms at the moment of oxidation hematite crystals are formed at the points of contact, binding the grains together. This results in increased strength of the pellet even at a relatively low temperature

(ii) If a surplus of air is present and the temperature is raised, an increased oxidation and stronger bond is attained. Ultimately the binding results mainly from recrystallization and grain growth of hematite in accordance with the second alternative shown in Fig. 1. The recrystallization of hematite occurs at 1100–1200° C., whereas the recrystallization of magnetite starts at 900° C.

(iii) If the amount of air is insufficient the oxidation can be delayed so much that at 900° C. magnetite is still present. On the surface of the single grains recrystallization and grain growth of the magnetite can take place in the same way as for hematite at higher temperatures. This can also occur if coal is added to the concentrate

(iv) At higher temperatures (1000–1200° C.) slag is formed from FeO and silica present in the gangue. The slag moistens the magnetite grains and delays a further oxidation. When the pellets are cooled the slag acts as a cement between the grains. This binding is not very strong, and it should be borne in mind that Fe_2O_3 does not react with silica at the temperatures used in pelletizing furnaces.

Another important investigation in this field has been published by Cooke and Stowasser.[16] Figure 2, which is taken from their paper, shows a hematite bond between pieces of magnetite which was obtained in the following way. Two flat-ground magnetite crystals were pressed together and heated to 800° C. in air for 2 hr. All along the seam hematite crystals were formed binding the crystals together. The experiment was repeated with two hematite crystals, but these fell apart after the heating.

When using very rich magnetite concentrates without mixing in coal or any other addition, the pelletizing process can be controlled in such a way that the pellets are completely oxidized and the bond is due to recrystallization of the hematite formed.

The mechanism of oxidation of magnetite balls has been studied by Zetterström,[17] who found that the following factors are important :

(i) Temperature
(ii) Velocity of the gas stream
(iii) Oxygen content of the gas
(iv) Grain size of the concentrate
(v) Porosity of the balls.

Zetterström found that oxidation takes place in two steps, in the first of which it is rapid and in the second slow. The speed and degree of oxidation rapidly increase with temperature. At 1000° C. and above the oxidation is increased by a high percentage of oxygen in the gas and also by a higher gas velocity. The speed and degree of oxidation increase with diminishing grain size and increasing porosity.

In the fired pellets concentric cracks are often found. These cracks diminish the crushing strength of the pellets. In the paper by Ilmoni and Tigerschiöld[13] it was emphasized that the concentric cracks are due to rapid drying, but Cooke and Ban[15] have found that similar cracks can also be formed at 1100–1200° C. in slowly dried pellets. The cracks then occur between the completely or partly oxidized outer layer and the unoxidized magnetite core. Ilmoni and Uggla[14] found that the strength of fired pellets depends on the rapidity of heating. For example, when the

Fig. 2—Line of cementation between two magnetite crystals clamped together, heated to 800° C. in air for 2 hr., and furnace-cooled × 2000

temperature was raised 72° C./min. the crushing strength in their experiments was only about 500 kg., whereas at slow heating, 8·3° C./min., the strength was 1250 kg. The oxidation at elevated temperatures occurs along a distinct concentric front, and between the recrystallized hematite and the still unoxidized magnetite core cracks can be formed as the magnetite, on recrystallization, shrinks from the surrounding shell. It is also possible that temperature differences between the outer layer and the core at certain moments can be so high that the formation of concentric cracks could be explained by stresses caused by differences in expansion. It is probable that the mechanism of formation of cracks can be explained by a combination of all these causes.

In practice it seems very difficult to avoid the formation of such concentric cracks in the balls. For obvious reasons a long drying time is difficult to obtain, and a double firing as recommended by Cooke and Ban will also make the process more complicated.

The Influence of Additives

Ilmoni and Uggla[14] have studied the influence of additives on the properties of green, dried, and fired pellets. To avoid the influence of the gangue a very rich concentrate from the Malmberget mine was used, which had the following analysis :

Fe_3O_4, %	99·17	P, %	0·005
SiO_2, %	0·16	S, %	0·004
Total gangue, %	about 0·83	Fe, %	71·8

The sieve analysis was as follows :

Mesh (Tyler)	Size, mm.	Accumulative, %
+ 65	+ 0·210	0·007
100	0·149	0·360
150	0·105	2·000
200	0·074	7·030
300	0·048	27·510
400	0·038	28·370
− 400	− 0·038	71·630

The specific surface of the concentrate was 1750 sq. cm./g. The definition and determination of the specific surface are obtained from the papers by Svensson[18] and Tigerschiöld and Ilmoni.[13]

As already mentioned, the properties of green, dried, and fired pellets and the resistance to rapid drying can be improved by certain additions. It is significant that the strength at reduction can also be appreciably improved in this way ; this property is of vital importance when using the pellets as raw material for the Wiberg sponge-iron furnace. A special test has been developed at Söderfors for determining the strength at reduction of the ores to be used. An amount of 500 g. of ore, crushed and sieved to a size between 10 and 16 mm., is reduced to FeO at 950° C. in a rotating tube furnace. The reduction is carried out only to FeO because it has been found that the ore is weakest in the FeO stage. The composition of the reducing gas must be adjusted so that only FeO is formed. At Söderfors the circulating gas from the Wiberg furnace is used, with the analysis : 50–58% CO, 12–18% H_2, 24–30% CO_2, and 2–5% H_2O. The furnace, which has an inner dia. of 130 mm., rotates at 66·6 r.p.m. After 3½ hr. the material is allowed to cool in a stream of nitrogen. The reduced material is sieved, and by experience it has been found that the amount of +10 mm. material should preferably exceed 80% of the total, and of +6 mm. material should exceed 90%.

In their investigation Ilmoni and Uggla[14] studied the influence of several additives. Of special interest are additions of bentonite, calcium oxide, calcium chloride, sodium chloride, and borax. Many other additives were also tried, *e.g.*, very fine ordinary clay, finely milled ash from oilbearing shales, waste pickle liquor, and sulphuric acid. More work remains to be done and is planned in this field. The most significant results are given in Table I, and the following conclusions can be drawn.

Bentonite—The additions of bentonite were 0·25% and 0·5% of a high-quality Wyoming sodium bentonite of the type used in foundries. The addition of as little as 0·25% of bentonite gave a marked increase in the crushing strength of the green balls. Bentonite addition also had a very good effect on the crushing strength of dried pellets and gave a better resistance to rapid drying. The crushing strength of fired pellets was appreciably increased and the shrinkage was higher. The reducibility of the pellets was not influenced by an addition of 0·5% of bentonite, and the strength at reduction was good.

Calcium Oxide—The addition of calcium oxide is of great interest. It is not very expensive and is of value when using the pellets in blast-furnaces as well as in basic open-hearth and electric steel furnaces. Additions of 0·5, 1·0, 3·0, and 5·0% of CaO were made. The resistance to dropping and knocking (repeated falls from a height of 4 in.) of the green pellets was appreciably increased and the crushing strength of the dried pellets was extremely good. The crushing strength of the fired pellets increased as the addition of lime increased, and this was also the case for the strength at reduction. The reducibility was somewhat diminished, but not to a significant extent.

Calcium Chloride—Calcium chloride has been used regularly in technical operation at Bodås and Söderfors. Trials made with 0·25 and 0·5% of $CaCl_2$ show that it has no influence whatsoever on the strength of the green balls but improves the crushing strength of the dried balls. The resistance to rapid drying is improved, and the strength of the fired balls is of the same magnitude as the strength when no additions are made. It also has a marked influence on the strength at reduction, and the reducibility is somewhat improved. Technical $CaCl_2$ is not very expensive and is easily dissolved in the moisture of the concentrate.

Sodium Chloride—An addition of 0·25% of sodium chloride has no influence on the properties of green balls and only a small influence on the crushing strength of dried balls, but it gives a very great improvement in the resistance to rapid drying. The crushing strength of the fired balls is increased slightly, but the strength at reduction is very low owing to a strong swelling action. On the other hand the reducibility is improved. The low strength at reduction makes it impossible to use this additive when the pellets are

used as raw material for the Wiberg sponge-iron shaft-furnace. For blast-furnace use, good reducibility is always desirable, but if a high percentage of pellets is used in the burden the low strength at reduction might be a disadvantage.

Borax—The addition of 0·15% of borax had no effect on the properties of green balls and very little on dry balls, but it had the same good influence as NaCl on the resistance to rapid drying. The strength of fired balls was not greatly improved, and borax had the same detrimental effect as NaCl on the strength at reduction.

No explanation has yet been found of the rather large variations in strength at reduction for highly oxidized pellets. Different lump hematite ores also show great differences in this respect ; hematite from the Steep Rock mine in Canada has a very good strength at reduction, whereas the Venezuelan hematites from El Pao and Cerro Bolivar disintegrate more or less at reduction. In these cases the difference in the analyses is very small.

If the percentage of free silica in the concentrate is low, an addition of lime results in the formation of calcium ferrite, and Ilmoni and Uggla[14] have found by X-ray examination that the ferrite is probably of the composition $CaO.2Fe_2O_3$. If, however, sufficient free silica is present the dominant reaction will be the formation of calcium silicate.

Ban and Erck[19] found that an addition of limestone markedly improves the strength of fired pellets. It is of interest that this addition is also advantageous when hematite concentrate is used as raw material. No trials on a large scale have been carried out to show the different effects of additions of lime and limestone. The CaO will combine with the moisture in the concentrate, which is an advantage if the moisture content is high. It must be assumed that the decomposition of the limestone in the pelletizing process will increase the fuel consumption. Moreover, an addition of limestone cannot be expected to influence the strength of dry balls, and this is a significant advantage when lime is added in the form of CaO or $Ca(OH)_2$.

When adding sodium chloride a ferrite also seems to be formed, but the actual composition has not yet been determined. At an early stage trials had been made in a pilot furnace using an addition of about 0·25% of NaCl, and it was found that the fired pellets contained all the sodium added to the concentrate. When trying to repeat the test in an electrically heated laboratory furnace, using the rich Malmberget concentrate, the sodium chloride did not stay in the pellets. An attempt was then made to adjust the atmosphere in the electric furnace to about the same as that in an industrial furnace, by adding certain amounts of carbon dioxide and water vapour, but the sodium chloride still evaporated. Finally, SO_2 was added to the atmosphere, after which all the added sodium combined with the iron oxide. When burning sulphur-free concentrates all the sulphur in the atmosphere comes from the fuel gases, but in this case the amount of sulphur in the oil used as fuel was not sufficient to form Na_2SO_4 from more than a small part of the sodium chloride added. A possible explanation of this phenomenon is that in the full-scale furnaces a circulation of SO_2 or SO_3 takes place within a certain temperature range. Thus, a probable formula for the formation of sodium ferrite is :

$$2NaCl + SO_3 + H_2O = Na_2SO_4 + 2HCl$$
$$Na_2SO_4 + Fe_2O_3 = SO_3 + Na_2O.Fe_2O_3$$

On adding NaCl as well as $CaCl_2$, hydrochloric acid is formed and condensation in the furnace stack must be avoided, to prevent corrosion. This precaution must be taken in any case, however, as the formation of SO_2 and probably of SO_3 from sulphur in the oil or in the ore always occurs.

In all the trials made by Ilmoni and Uggla the additives were mixed very thoroughly with the concentrates before rolling. In practice the additives are usually added on the top of the layer of concentrate on the transportation belt to the balling drum. Naturally, a mixing occurs in the drum, but experiments have shown that much can be gained by more thorough mixing. Ilmoni and Uggla found that if the adequate amount of bentonite is, for example, 0·3% when thoroughly mixed, an amount of 0·5% is necessary if the mixing is made in accordance with general practice. In other words, according to this experiment about 40% of bentonite could be saved by more efficient mixing.

If CaO is used as an additive, the investigations show that even with thorough mixing the mixture should be stocked for 1–2 hr., to allow the CaO to react with the water of the concentrate to $Ca(OH)_2$. If the rolling is made immediately after the lime addition, the dried as well as the fired balls are of poor strength. Thus, the lime should be added to the filtered concentrate in the concentrating plant, as it will then have ample time to react with the water during transportation to the pelletizing plant and in the storage bins. If soluble salts such as $CaCl_2$, NaCl, or borax are used as additives it seems unnecessary to make a thorough mixing. After a short time the salts are dissolved in the water present in the concentrate, and a good distribution is achieved.

The Influence of Ball Size

In the U.S.A. the size of the balls has been kept mainly at about $\frac{7}{8}$ in., whereas in all Swedish plants hitherto in operation the ball size is about $1\frac{1}{4}$ in., because the existing Wiberg sponge-iron furnaces are dimensioned for a not-too-high pressure drop of the circulating gases in the furnace. Ilmoni and Uggla have investigated the influence of ball size on the properties of green and fired pellets and have drawn the following interesting conclusions :

(i) The ball size has a marked influence on the output from the drum. This conclusion is strengthened by the fact that the big balls crush the smaller balls when tumbling in the drum

(ii) If D denotes the ball diameter, the crushing strength of green and dried balls is directly proportional to about D^2. This means that small and large balls can withstand the same load in the furnace, and that when using large balls the zone of high temperature can be kept at the same level in the furnace as if smaller balls were charged

(iii) The resistance to rapid heating is only slightly influenced by the ball diameter

(iv) The resistance to knocking for fired balls (repeated falls from a height of 5 ft.) is proportional to about D^{-2}

(v) The time of oxidation and reduction is proportional to D^2 and thus to the surface area of the pellet

(vi) The time for the exchange of heat from a hot ball to a stream of cold gas is proportional to about $D^{-1 \cdot 4}$

(vii) If a gas stream passes through a column of balls, the pressure loss at the gas velocities used in pelletizing furnaces is proportional to about $D^{-1 \cdot 13}$. This power would be about -2 if the balls were polished. The surface roughness of the balls makes the pressure-loss difference between large and small balls lower than would be expected.

The above results show that there are strong reasons for making small balls in the pelletizing furnaces ; the production can be kept at a higher level and the fired pellets will be stronger. It also seems feasible to reconstruct the Wiberg shaft-furnaces for the use of smaller balls than are now used.

Pellets to be used as feed ore in open-hearth and electric furnaces should preferably be large. It is essential that the individual balls should be heavy enough to overcome the surface tension of the slag so that they can sink through the slag to the steel bath. When using pellets as feed ore in converters, much time can be saved if the pellets can be charged against the blast into the upright converter. Balls of $1\frac{1}{4}$-in. dia. can successfully be charged in this way. For blast-furnace use the ball diameter could be smaller than $\frac{7}{8}$ in. The minimum diameter of pellets to be produced in shaft furnaces has not yet been thoroughly investigated, but the Americans probably have good reasons for the size they now use.

Comments on Furnace Design and Operation

On studying the details of the pelletizing process it seems surprising that it can be performed in large-scale shaft furnaces. It is a bold and clever idea to charge green balls into a shaft furnace, and to dry and heat them rapidly under an increasing load to a temperature which must be high enough to make all the grains in the balls bake together, but not so high that the individual balls stick to each other. Working between this "Scylla and Charybdis" necessitates very exact temperature control and a most uniform gas and heat distribution at every level of the furnace.

There are still different opinions about shaft-furnace design, and both round and rectangular furnaces have been tried in full-scale operation. It is obvious that width is a very important measure in a furnace heated by fuel gases entering from combustion chambers outside the furnace ; according to present experience the width of the furnace should not exceed 6 ft. The length of a rectangular furnace will be limited by masonry and reinforcement problems. A round shaft is a sturdier design, but the diameter of the furnace cannot be increased above a certain value.

It is obvious that it is much easier to control a small furnace than a large one since the gas and heat distribution are greatly influenced by the furnace dimensions, as also is the minimum size of balls that can be used.

An interesting experiment was made recently by Ilmoni and Uggla, to determine the sticking tendency of balls made from different Swedish concentrates. The tests were carried out as follows. Two carefully rolled and dried, but not fired, 20-mm. dia. balls were inserted in a vertical tube furnace, which was kept at a high temperature. At 5 min. after the charging a load of 1 kg. was applied on the upper ball, and after 10 min. the load was removed and the balls were taken out. There was a very great difference in sticking tendency between the concentrates tried. The sticking temperature of the very pure Malmberget concentrate was as high as 1400° C., whereas Bodås balls stuck at 1250° C. An addition of 0·5% of bentonite seemed to improve the results from the Malmberget concentrate ; the temperature of the furnace could be raised to 1450° C. before serious sticking between the balls occurred. An addition of 0·4% of $CaCl_2$ plus 1% of CaO raised the sticking temperature of the Bodås balls, whilst an addition of 3% of CaO to the Malmberget balls lowered the sticking temperature by about 50° C.

It is obvious that the amount of gangue and its composition, and also the additions made, have a significant influence on the sticking properties of the balls when these are baked in the pelletizing furnace. If the margin between the temperature for making the grains grow together and the temperature at which individual balls stick together is wide, the difficulties in controlling the furnace will be much less than if this margin is narrow.

It is essential that the green balls are carefully charged into the furnace and that the distribution of the pellet layer is made in the right way. If the green balls are weak, a formation of fines during transportation and charging is unavoidable. The presence of even a small amount of fines makes it difficult to obtain regular and even heat and gas distribution in the furnace.

The charging of green pellets and the discharging of fired pellets must be made continuously, so that the upper layer in the furnace can always be kept at the same level. To avoid the serious troubles involved by sticking of the charge it is advisable, in most cases, to install mechanical breakers at the bottom of the furnace. The maximum temperature necessary to produce strong baked pellets varies, of course, with the composition of the concentrate and the additions made. For the rich Malmberget concentrates the temperature can be kept above 1250° C., whereas concentrates with lower iron contents cannot withstand a higher maximum temperature than 1150° C.

The furnace output depends to a large extent on the size of the balls and the specified properties of the pellets to be made. Pellets for making sponge iron in the Wiberg shaft furnaces must have a high strength at reduction. For use as feed or charge ore in open-hearth or steel furnaces the pellets should be hard-burned and fully oxidized, whilst specifications for blast-furnace use can be less rigid. Hitherto in Swedish pelletizing plants, mainly very hard-burned strong pellets have been produced and the size of the pellets has been about $1\frac{1}{4}$ in. In this case the production per sq. ft. of furnace area must be kept at the rate of 2·5–3 metric tons per day, whereas in

America productions of over 5 tons per sq. ft. per day have been reached in regular operation with $\frac{7}{8}$-in. pellets.

A drop test and an abrasion test are standardized in America for judging the quality of the fired pellets. In the drop test 50 lb. of pellets are dropped through a 32-ft. high, 6-in. wide tube against a thick steel plate. After 5 drops the pellets are sieved and the quantity of −10 mesh is weighed. If this quantity is less than 10%, the pellets are considered to be strong enough to withstand the handling and transportation from the mines to the blast-furnace plants. When making the abrasion test, 50 lb. of pellets are inserted in a drum with a dia. of 36 in. and a length of 18 in. In the drum a $2\frac{1}{4}$-in. high lath is applied which lifts the pellets and drops them when the drum is rotated. The drum is closed and is then rotated 200 times at 24 r.p.m. The pellets are sieved and the quantity of −28 mesh is weighed. If this is 20% or more the pellets are too weak.

High-quality $1\frac{1}{4}$-in. Swedish pellets of the Malmberget concentrates have been tested by both these methods. In the drop test, only 4·9% fines of −10 mesh were obtained, and in the abrasion test the quantity of −28 mesh was only 7·1%.

Heat Balance for the Shaft-Furnace Pelletizing Method

All industrial pelletizing furnaces hitherto constructed are heated with fuel oil. When rich magnetite concentrates are used the oil consumption varies between 1·2 and 2·0%, depending on the design and size of the furnace.

Under the supervision of L.O. Uhrus a very careful investigation of the heat consumption in a pelletizing furnace was recently made at the small S.K.F. plant at Mossgruvan. The following heat balance is typical for the process :

	Mcal.	%
Heat Input per 1000 *kg. of Pellets*		
Combustion heat of fuel oil (14·63 kg.)	146·59	55·9
Heat from oxidation of Fe_3O_4 and S in the concentrate	105·89	40·4
Sensible heat in the concentrate (1060 kg.), oil (14·63 kg.), and air (987 cu. m. at N.T.P.)	9·56	3·7
Total	262·04	100·0
Heat Output per 1000 *kg. of Pellets*		
Sensible heat in waste gases (1110 cu. m. at N.T.P.) at 320° C.	114·73	43·8
Combustion heat in waste gases	3·37	1·3
Sensible heat in 1000 kg. of pellets at 205° C.	35·07	13·4
Vaporization of 85·4 kg. of water at 0° C.	51·67	19·7
Heat losses through furnace walls	57·20	21·8
Total	262·04	100·0

The composition of the concentrate and the pellets was as follows :

	Concentrate	Pellets
Fe_2O_3, %	66·500	96·340
FeO, %	29·800	0·500
S, %	0·030	0·003
P, %	0·004	0·004
Gangue (rest), %	3·476	3·153
$CaCl_2$ (added), %	0·190	—
	100·000	100·000
Fe_{tot}, %	69·7	67·8
Oxygen in iron oxides, %	26·5	29·0
Degree of oxidation, %	88·9	99·8

At a trial made in 1951 at Mossgruvan, using Malmberget concentrates, the following analytical results were obtained :

	Concentrate	Pellets
Fe_2O_3, %	69·000	98·000
FeO, %	30·100	1·140
S, %	0·004	0·004
P, %	0·009	0·010
Gangue (rest), %	0·887	0·846
	100·000	100·000
Fe_{tot}, %	71·7	69·5
Oxygen in iron oxides, %	27·4	29·7
Degree of oxidation, %	89·1	99·6

As seen from the above analyses, the pellets made from Swedish concentrates have extremely high contents of iron and oxygen and very low contents of S, P, and gangue.

DEVELOPMENT OF PELLETIZING IN THE U.S.A.

As early as 1920 it was recognized that the resources of rich hematite Mesabi ores were limited, and in 1923 a pilot plant for concentrating and sintering was in operation at Babbit, Minnesota, for the beneficiation of lean taconite ores in this district. To-day many of the ore companies have to face the fact that their hematite mines will be exhausted within a relatively short time. Thus the possibility of utilizing the huge deposits of taconite with 25–30% Fe has become a problem of the utmost importance.

It was soon found that the lean Mesabi taconites must be milled to a high degree of fineness before a magnetic concentration can be efficiently made, so that the pelletizing process is looked upon as a key solution to the taconite problem. The big mining companies in the Mesabi district, particularly the Erie Mining Co. and the Reserve Mining Co., have spent enormous sums of money on large-scale pelletizing trials and have now decided to build plants for many million tons. The Erie Mining Co. has recently announced[20] that a plant will be erected near the pilot furnaces at Aurora to give a yearly production of $7\frac{1}{2}$ million tons ; this may eventually be extended to give a pellet production of $10\frac{1}{2}$ million tons. The investment cost of this plant will exceed $300 million. This will include mining facilities, milling and concentrating plants, pelletizing equipment, and new railroads for the transportation of the product. The Reserve Mining Co.,[21] which has been running pilot plants at Ashland and Babbit, will build their full-scale plant at Beaver Bay on Lake Superior. It will be dimensioned for a yearly production of $3\frac{3}{4}$ million

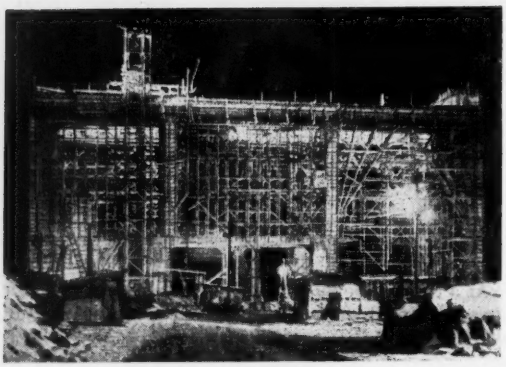


Fig. 3—The Malmberget pelletizing plant under construction (photograph taken 5th December, 1953, at 2 a.m.)

tons and will later be extended to produce 10 million tons. The Bethlehem Steel Co., which has successfully run a pilot plant at Lebanon, has recently extended it to give a yearly production of about 300,000 tons. They are now building a plant in Canada for a production of 500,000 tons annually, and plan within a few years to produce 2 million tons of pellets per year at a new mine at Morgantown, Pa. The Cleveland-Cliffs Iron Co., which has run a small pilot furnace at Ishpeming, is now putting up a pelletizing plant to produce about 600,000 tons per year, for the agglomeration of floated hematite concentrates ; a specially built sintering machine designed by the Koppers Co. in Chicago will be used for the pelletizing. The International Nickel Co. at Sudbury, Canada, is planning a pelletizing plant for the agglomeration of large quantities of pyrite residues. In this case a sintering machine will probably be used.

The undoubted success of these projects is due to the work done by people such as E. W. Davis, C. V. Firth, P. L. Steffensen, and F. D. DeVaney. The fundamental investigations in the U.S.A. on furnace design and operation should be reported by the American pioneers and by no one else.

SWEDISH PELLETIZING PLANTS

As mentioned earlier, the small pilot plant at Sandviken was erected as early as 1947, and in 1950 the S.K.F. Co. built another pilot furnace producing 25 tons per day at their Mossgruvan mine. This furnace has been operated continuously and the product has been used as feed ore in the open-hearth and electric steel furnaces at Hofors. The furnace is rectangular, with an area of 10 sq. ft. This plant was erected so that the S.K.F. Co. could gain experience for the construction of a pelletizing plant at Hofors, which will be used for the production of pellets for making sponge iron. The furnace at Hofors will be dimensioned for a production of 150 tons per day and will come into operation in the autumn of 1954.

Another small pilot plant, belonging to Reymersholms Gamla Industri AB, was built in 1953 at Kopparverket, Hälsingborg. This plant has been used exclusively for making pellets from pyrite residues. The results are said to be good, but no official report is available.

The first industrial pelletizing plant in Sweden was started in 1952 by the Sandvikens Jernverks AB at their mine at Bodås. This plant is built for a production of 120 tons per day. The furnace is rectangular and consists of three shafts, each 3 ft. 4 in. by 4 ft., built together within the same shell. The product is regularly used at the sponge-iron furnace at Sandviken, with very good results.

At Söderfors a third plant came into operation in December, 1952. The furnace is round with a diameter of 5 ft. 3 in., and the rated production is about 55 tons per day. The sponge-iron plant at Söderfors is now using only pellets as raw material. The concentrate used comes from the Ramhäll mine near Dannemora.

In 1953 a sponge-iron plant and a pelletizing plant were started at Hällefors. The pelletizing plant is dimensioned for a production of about 50 tons per day. The furnace is round and has a diameter of 4 ft. 1½ in. The raw material used is mainly concentrate from Persberg. A second furnace is planned for a production of about 100 tons per day.

Quite recently a fifth pelletizing plant was started at Persberg, where the Uddeholm Co. has built a sponge-iron plant. The pelletizing furnace is rectangular and has one shaft with an area of 22 sq. ft. Persberg concentrate is used as raw material.

At present the Luossavaara-Kiirunavaara AB is erecting a pelletizing plant at Malmberget (Gällivare) in Lapland (*see* Fig. 3), which is planned to be in operation by the autumn of 1954. The furnace unit, which is the biggest hitherto constructed, consists of two shafts, each 6 ft. by 10 ft., within the same shell. The rated production of 1¼-in. pellets is 360 tons per day. If smaller balls are made the furnace should, according to American experience, be able to produce about 600 tons per day. In this plant the very rich Malmberget concentrate will be used as raw material. The product is meant for export, and will probably be used mainly as feed ore in steel furnaces. If the plant runs successfully and the demand for the product is satisfactory, the plant will be extended within the next few years to give an annual production of 600,000 –800,000 tons.

According to this survey five industrial pelletizing plants are running, including the pilot plant at Mossgruvan, which is in regular operation. Before the end of 1954 seven pelletizing plants will be operating in Sweden, with a rated production of about 250,000 tons per year.

Most of the mechanical equipment for the pelletizing plants in Sweden has been designed and delivered by the Sala Maskinfabriks AB at Sala, Sweden. A detailed description of these plants and the practical experience gained has not been given here, but it is hoped that the representatives of the different companies will report some of their results in discussion.

The unquestionable success of the Swedish pelletizing plants hitherto in operation is due to extremely good team work, in which fundamental knowledge obtained from laboratory work has been utilized for the technical developmentof the process. The different companies have exchanged experiences and have

co-operated openly and efficiently. The Sandvikens Jernverks AB, which is one of the pioneers in this field, permitted trials to be made in the pilot furnace at Sandviken and also in the full-scale plant at Bodås. The S.K.F. Co. has also been very helpful in trying out different concentrates in their pilot furnace at Mossgruvan. By continued co-operation it is hoped to achieve still better results and to obtain a more complete knowledge of the process. The contacts that have been made with the American specialists in this field have also been of the utmost value. The mere fact that furnaces for daily productions of 400 tons and more are in continuous operation, and the disclosure of the American plans for treating millions of tons of taconite concentrates, has strengthened Swedish confidence in the pelletizing method.

PELLETIZING PLANTS IN OTHER EUROPEAN COUNTRIES

In 1930 Brackelsberg[22] tried a pelletizing process at Rheinhausen, in Germany ; his pellets were not fired, and he used sodium silicate as a binder. In 1950 and 1952 Sengfelder[23, 24] reported work on the production of pellets from Doggererz concentrates. A small pilot furnace was built by the Friedrichshütte at Herdorf, Siegen ; quite good results were achieved, despite the fact that the ore contained 12–14% of chemically bound water. For reasons which are not clear, the experiments have not led to the construction of a full-scale furnace.

At the Imatra plant of the Vuoksenniska Co., in Finland, Holmberg has developed a process for recovering zinc, copper, and cobalt from pyrite residues where at the same time pellets are produced. No results have yet been published.

As reported by Ridgion,[25] pilot-furnace trials have been made in England on the pelletizing of hematite concentrates and pyrite residues. In Norway, laboratory experiments have been made by the Sydvaranger Co. at Kirkenes. This company has decided to build a full-scale, American-type pelletizing plant in the near future.

OTHER PELLETIZING METHODS

The difficulties of controlling the shaft-furnace pelletizing process have resulted in many proposals for other equipments, but these have not yet been tried sufficiently in full-scale operation. An obvious solution is to adopt the continuous sintering machine of the Dwight–Lloyd type for baking the green balls into pellets. Nearly all the firms now constructing Dwight–Lloyd machines are working on this problem. In America, the Allis Chalmers Co. and the Arthur G. McKee Co. are co-operating in designing a sintering machine for this purpose.[26] In February, 1954, the first full-scale machine was put into operation at the Reserve Mining plant at Babbit, Minnesota. For some time the Lurgi Co. has been making laboratory experiments in Frankfurt, and it is now operating a pilot plant producing 25 tons per day at Watenstedt, in Germany.

It is too early to give an opinion on the possibilities of this method. The problem of sticking, which can be a serious drawback in a shaft furnace, is obviously of small consequence in a sintering machine. If the margin between the sintering temperature and the sticking temperature is small, the sintering machine should probably be considered. The heat consumption, however, must be higher, and the investment cost for such plant, as well as the repair costs, will probably exceed the corresponding figures for a shaft furnace. The pellets hitherto produced in sintering machines have been of a much smaller size ($-\frac{1}{2}$ in.) than shaft-furnace pellets, and it is probably difficult and also uneconomical to produce large pellets in such machines.

In a paper read at Cincinnati in 1950, Cavanagh[27] reported on work done on the extrusion of fine ores into sausage-like pieces which, after a short drying or hardening process, could be charged directly into the blast-furnace. The present author suggests, however, to avoid confusion, that this method should not be referred to as 'pelletizing.' This term should be reserved for methods where balls are formed by rolling moist concentrates in a drum or similar equipment. The extrusion method has been tried on a large scale in the United States for ores containing clay. Waste pickle liquor has been used as binder. Very little information on the actual use of this method has hitherto been available in the U.S.A., but Stirling[28] has now published valuable information on the work done at Corby, in England. For certain ores this method might be a useful complement to the sintering and pelletizing methods in the efficient preparation of blast-furnace burden.

POSSIBLE FUTURE OF THE PELLETIZING PROCESS

The future of the pelletizing process depends on the possible saving in production cost in comparison with other methods, and also on the value of the product for use in the iron and steel industry. According to Swedish experience the cost of pelletizing in the moderate-size units hitherto used is not higher than that of sintering carried out by the classical methods. In some cases pelletizing will be less expensive than sintering ; *e.g.*, when the available concentrate is very finely milled. The production from ordinary sintering machines will be decreased when treating a fine mesh material, partly because the amount of returns will be much higher than usual.

The decision to build the huge plants in the U.S.A. and Canada is based on the conviction that the pelletizing method is the best so far found for agglomerating finely milled magnetite concentrates, and also on the experience gained when the pellets were tried in blast-furnaces and as charge and feed ore.

In Sweden large resources of magnetite ore are available which in most cases can easily be concentrated to an iron content of over 60% without grinding the product to a high degree of fineness. Thus, most of these concentrates can be sintered effectively for blast-furnace use according to the classical methods. An exception is the Malmberget concentrate, which must be very finely ground when efficient removal of phosphorus is required.

The original reason for Swedish interest in the pelletizing method was the apparent possibility of producing a suitable raw material for the Wiberg

sponge-iron furnaces. To achieve the desired high iron content (68% and above) the concentrates have to be milled to about the same fineness as the Mesabi ores. As reported earlier (p. 22), the necessary agglomeration has been successfully achieved by applying the pelletizing method.

This method, at least when large shaft-furnaces are used, seems to be less suitable for the agglomeration of hematite concentrates and pyrite residues; like most new methods its use will be limited to the treatment of certain types of ore, and it will be a useful complement to the beneficiation of the iron ore resources.

If for some reason the properties of the pellets are superior to those of ordinary sinter, the method will probably prove sufficiently economical even when extra grinding of the concentrate must be done to make pelletizing possible.

So far, no information has been published on the results achieved when using pellets in blast-furnaces and in steel furnaces. From reliable sources in the U.S.A., however, it is understood that blast-furnace operators who have once tried pellets insist on always using them. This is understandable in view of the high dust losses occurring when unscreened Mesabi ores are used, and the pellets also are superior to the hard-burned sinter common in the U.S.A. In their case an addition of highly oxidized rich pellets must give a considerable saving in fuel consumption and also in increased production.

At Sandviken and Hofors, only pellets are now used in steel furnaces, and very good results have been achieved. The pellets are richer in iron than the magnetite lump ore previously used, and the amount of oxygen in the ore is increased. Pure hematite contains 8·7% more oxygen than magnetite so that, for example, the rich Malmberget pellets with 69% Fe will contain 14·1% more oxygen than the best 'Kiruna A' magnetite lump ore with 68% Fe. Well-burned pellets will stand handling and transportation without substantial cracking or abrasion, and should be an ideal charge and feed ore in all types of steel furnaces. The porosity of the pellets is 20–25%, the apparent density about 3·95, and the bulk density about 2·2 tons/cu. m. (137 lb./cu. ft.).

Most pelletizing plants hitherto constructed are placed at the mines. This is an advantage when the mine is located in districts where freezing makes it impossible or difficult to transport moist concentrates in the winter. The dead freight for the moisture content of the concentrate is also saved if the pelletizing is done at the mine. On the other hand, the specifications for the pellets would be less rigid if handling and transportation of the product could be avoided as much as possible. This is achieved if the pelletizing furnaces are placed at the steel plants.

Acknowledgments

The author wishes to emphasize that the work done in Sweden is due to the excellent co-operation between the members of the Committee formed by Jernkontoret. Many of its members have made outstanding contributions to the design and operation of the pelletizing method, as well as to theoretical knowledge in this field.

The author thanks Jernkontoret and the Grängesberg Corporation for their permission to publish this paper.

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The Pelletizing of Northampton Sand Ironstones by Vacuum Extrusion

THE EXPERIMENTAL AND PILOT PLANTS AT THE CORBY WORKS OF STEWARTS AND LLOYDS LTD.

By A. Stirling, B.Sc., A.R.T.C., F.I.M., M.Inst.F.

SYNOPSIS

The essentials of the vacuum-extrusion pelletizing process are outlined and its application to the ironmaking practice based on low-grade Northamptonshire ores, at Corby, is stated. The equipment of an experimental and a (later) pilot plant are described and details are given of their operation in relation to the physical properties of the raw materials and the products. The enrichment of the Northamptonshire ore pellets by additions and by high-temperature firing is discussed and the conditions necessary for sulphur removal are given. In a concluding section the development of the process at Corby is assessed. 930

THE VACUUM-EXTRUSION PROCESS

THE vacuum-extrusion process is not yet widely used for the agglomeration of iron ores. There are a few post-war installations in the United States of America with capacities of up to 50 tons/hr. for processing mixtures of iron ores and flue-dust, or flue-dust alone, and the Corby plants—which it is the purpose of this paper to describe—are, as far as the writer is aware, the only vacuum-extrusion installations in Europe. The current interest in pelletizing, as Bell has pointed out,[1] has been stimulated by the increasing proportion of fines in the ores available and also by the need for an inexpensive method of agglomerating beneficiated ores whether from imported or native supplies.

There are several examples of the successful application of vacuum extrusion in the chemical trades, and many, of course, in the ceramic and refractories industries where de-airing has increasingly become a principal manufacturing process over the last twenty years.

Clay-working machinery has been used as the starting-point of the process as applied to iron ores but modifications to the standard machines have been necessary in most applications. These modifications are designed to increase strength since ores, and ore-flue-dust mixtures, are less plastic and more abrasive and granular than clays and require a greater power input per unit of product.

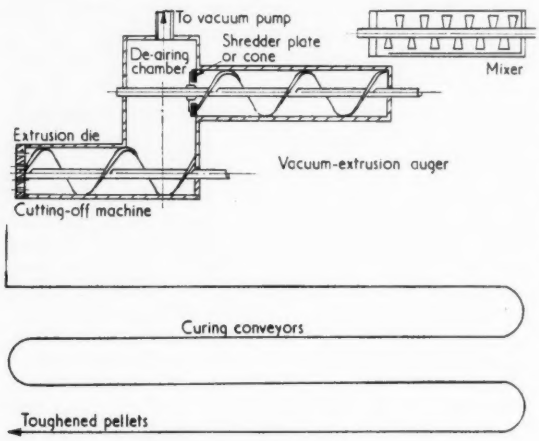

Fig. 1—Diagrammatic sketch of vacuum-extrusion plant

The essentials of a vacuum-extrusion plant for iron ores are shown in Fig. 1, which indicates diagrammatically the layout of the pilot plant installation at Corby. The ore is subjected to vacuum in a de-airing chamber, which is sealed on the inlet side by a cone, or perforated shredding device, and on the outlet side by the die itself. While columns of a variety of profiles can be extruded separately or simultaneously, the most convenient shape for the pellet is a normal cylinder. In some of the American plants the extruded columns are seemingly 'short' enough to break off, under their own weight, into suitable lengths but with more plastic ores, a cutting-off machine is necessary. The subsequent handling of the extruded pellets depends on the properties of the ores being agglomerated, the extrusion practice in terms of the use of binders and other factors, but the obvious aim is to produce self-hardening pellets which become good blast-furnace burden material in the shortest possible time.

With so few known applications of the process to the extrusion of iron ores, the literature is not extensive. The first scientific study of the subject was initiated in Canada by the Ontario Research Foundation

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Table I

BLAST-FURNACE PRACTICE, 1953—AVERAGES

The average iron analysis was: Si 0·95%; S 0·237%; Mn 0·85%; P 0·209%

Furnace	No. 2	No. 1	No. 3	No. 4
Hearth dia., ft.	19·5	20	20	20
Working volume (C.L. tuyeres to 6 ft. below big bell closed), cu. ft.	20,170	27,161	23,692	26,601
Burden, lb./ton of iron				
Lump ore (including pellets)	3,791	3,500	3,377	3,257
Sinter	2,094	2,039	2,258	2,059
Fluxes and Scrap	816	790	816	804
Total	6,701	6,329	6,451	6,120
Coke, lb./ton of iron	2,181	2,027	2,028	2,028
Slag, lb./ton of iron	1,858	1,745	1,754	1,771
Iron, tons/ day	419	473	468	478
tons/day/sq. ft. of hearth	1·40	1·51	1·49	1·52
tons/day/100 cu. ft. of working volume	2·02	1·74	1·98	1·80
Coke, tons/day	408	428	424	433
tons/day/sq. ft. of hearth	1·36	1·36	1·35	1·38
Slag Analysis, %				
SiO_2	33·5	33·8	33·8	33·6
Al_2O_3	22·5	22·7	22·6	22·7
CaO	33·5	33·5	33·5	33·7
MgO	6·5	6·4	6·6	6·6
MnO	1·4	1·4	1·3	1·3
S	1·4	1·4	1·4	1·4
FeO	1·2	1·1	1·1	1·1

and reported[2] by Cavanagh in 1950 and, in 1952, Ludwig reviewed[3] the place of vacuum extrusion in the minerals agglomerating industry of the United States.

Cavanagh has explained the initial interest of the Ontario Research Foundation in the vacuum process while investigating the production of sponge iron from ore bricks made from −8 mesh (Tyler) hematite. There were indications that these hematites could, without burning, be processed to good blast-furnace burden material. It was concluded in this work that the largest practical size for extruded pellets is a diameter of 3 in. and it was added that, after considerable experimentation, a method was devised of rolling the pellets down an inclined trough to round off their corners.

In this Canadian research it was found that some hematite ores of a clay-like nature could be extruded without the addition of a binder. Where the size grading of the material to be extruded was not initially suitable, or where the ore was not sufficiently plastic, the addition of fine, plastic ore was found to help and such unpromising materials as taconite concentrates and mill scale were successfully pelletized by this method. Otherwise, the addition of a binder may be necessary and Cavanagh adds that there is no single binding material which will give the combination of the desired properties in pellets which are not to be burned before reaching the blast-furnace. Ludwig also comments on the complexity of the search for the ideal binder.

Some of the features of successful practice which have been emphasized in these two papers have emerged with the same importance from the Corby investigations. The need for an auger design and an availability of power for its operation suited to the nature of the raw materials is the first essential. Further, an adequate mixing and pugging of the raw mixture and efficient de-airing are necessary and, in general, the pellets should emerge from the extrusion die hot and steaming.

With this measure of agreement, this account of the Corby experiments, which began in 1950, departs from the work described by Cavanagh and by Ludwig. Because of the peculiarities of the Northamptonshire ores so far investigated, no binders are necessary and the aim at Corby has been to process rather coarse run-of-plant ore fines without partial drying and without fine grinding.

APPLICATION OF THE PROCESS AT CORBY

The Corby blast-furnaces[4] are part of an integrated iron, steel, and tube works and supply Thomas iron for an annual output of about 600,000 tons of basic Bessemer steel. The basis of the ironmaking practice is the smelting of ores from the Northampton Sand Ironstone bed[5] which is characterized by comparatively high phosphorus and low iron contents. These ores are blended to produce a slag having a lime/silica ratio of approximately unity, which is characteristic of acid-burdening practice.[6] The ore is mined by the opencast operations which were described[7] in the Presidential Address to The Iron and Steel Institute in 1950, and is prepared[8, 9] by crushing to 2½ in., bedding by the Robins-Messiter system, and by screening, on gas-heated cloths, at ⅝ in. The −⅝-in. fines

fraction is usually from 35 to 40% of the total, depending on the nature and the moisture content of the ores, and screening is often difficult.

The furnace practice, as given by the annual averages for 1953, is indicated in Table I. The weight of pellets charged during the year was 4800 tons ; as pellets were used only in the latter weeks this amount, being small on the annual basis, has been included with the usage of lump ore. The average chemical analyses of the lump ore, ore fines, sinter, coke, and the furnace burden (including the flue-dust and the silicon requirement of the hot metal in terms of silica but not the coke ash) are given in Table II. It is evident that the lump ores are closely balanced in their silica and lime contents but neither the ore fines nor the sinter is so balanced. Each is siliceous and, on the as-fluxed basis, their iron contents amount only to 27·2% and 37·0%, respectively. The burden analyses are notable for their similarity and this is true of the furnace practice in general terms (Table I) which demonstrates the close identity of operation attainable by modern methods of ore preparation.

The ores fines—approximately $\frac{3}{4}$ million tons per year—are sintered in up-to-date Dwight Lloyd machines, but the clay-like and sticky nature of the ores makes difficult the application of measures to control sinter quality and even hazards the regularity of plant operation, although the recent introduction of a rotating-shell ore storage bin[9] has done much to ensure an even supply of ore to the strands.

Even so, there remain considerable operating problems in sintering these ores and, under present-day conditions, the process has become costly to an extent which may be approaching the limits of its advantages. Further, the installation of sintering strands now involves not only large capital outlays but also such operating charges as the cost of suppressing the inevitable dust nuisance in locations where this is a social problem. The nature of the Northamptonshire ores suggests their agglomeration by a compaction process, but preliminary experiments on nodulizing and briquetting indicated that not only would drying and fine-grinding treatments be involved, but also that the agglomerates would not have the necessary green strength for handling to the blast-furnaces. On the other hand, it seemed from the very first attempts to apply vacuum extrusion to these ores that extensive pre-preparation would not be necessary and also that green pellets of sufficient strength could be produced involving only a simple curing process following extrusion.

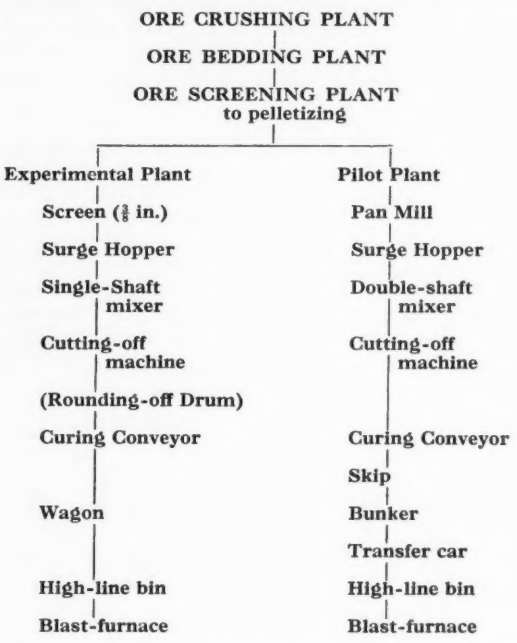


Fig. 2—Flowsheet of experimental and pilot plants at Corby

Table II

PERCENTAGE CHEMICAL ANALYSES

(As Charged to Furnaces)

	Lump Ore	Ore Fines	Sinter	Coke	Burden			
					Furnace			
					1	2	3	4
Fe	30·3	28·9	39·9·	1·5	35·8	34·6	35·5	35·5
Mn	0·2	0·2	0·3	—	0·6	0·6	0·6	0·6
S	0·27	0·19	0·12	1·00	0·20	0·20	0·20	0·20
P	0·7	0·7	0·8	—	0·7	0·8	0·8	0·8
SiO_2	8·3	9·7	13·3	5·0	9·3	9·3	9·5	9·4
Al_2O_3	5·4	5·7	7·9	2·6	5·7	5·7	5·7	5·7
CaO	8·2	6·7	9·1	0·4	9·6	10·1	10·0	10·1
MgO	1·3	1·3	1·6	0·2	1·9	1·9	1·9	1·9

Vacuum-extrusion pelletizing incurs, almost certainly, a considerably lower capital cost per unit of output than sintering and the operating costs also may well be lower. These advantages, coupled with the production of a uniformly sized agglomerate, make the vacuum process attractive at Corby. It is true that the pellets being, in effect, synthetic lump ore, they will reach the blast-furnace containing the volatiles which are normally removed by sintering but an interest in unfired pellets does not preclude the development of a process for the calcination of lump ore. The view is increasingly being held that efforts towards the beneficiation of iron ores should be extended to include as many of the ingredients of the burden as possible, and in the vacuum-extrusion process there are the possibilities of agglomerating fluxes, enrichers (including high-grade ores), and carbon-bearing materials with Northamptonshire ores as a base.

EXPERIMENTAL PLANT

The plant was started up in July, 1951 following preliminary trials with vacuum extrusion in October, 1950. The initial layout has, very largely, been retained excepting that, towards the end of 1951, an improved curing conveyor was installed.

Screen and Mixer

Before entering the mixer (Fig. 2) the ore is screened at $\frac{3}{8}$ in. on a double-deck Gyrex screen 54 in. long by 24 in. wide. Choking of the screens is minimized by gas heating and the screened ore is conveyed

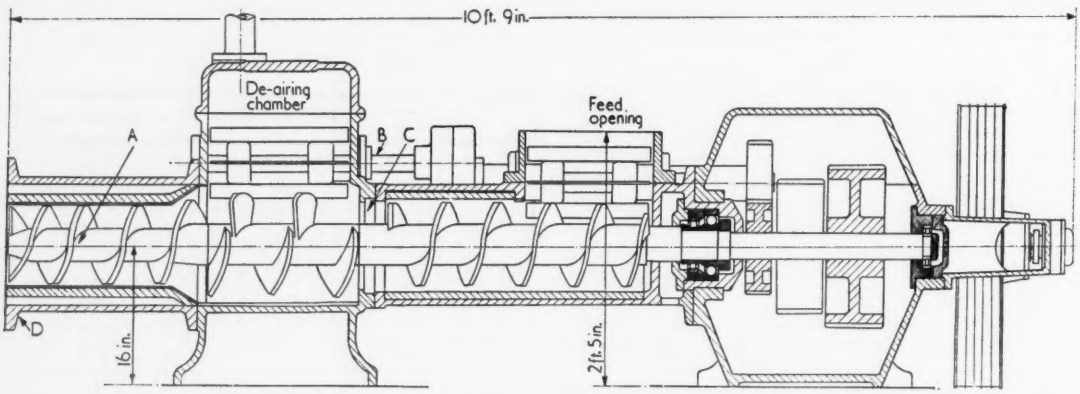


Fig. 3—The 9in. de-airing auger: (*A*) Main shaft; (*B*) Packer shaft; (*C*) Shredder plate housing; (*D*) Flange for die

to a surge hopper which gives a regulated feed to a single-shaft mixer which is 7 ft. 6 in. long by 18 in. wide and has a curved bottom concentric with the shaft.

The 36 steel mixer-blades (6½ in. long by 5 in. wide and ¾ in. thick) were built up on their peripheral edges with hard facing alloy when necessary to compensate for wear. The throughput of the mixer between these treatments was about 600 tons of ore. The best setting of the mixer blades was found by experience and was such that the rate of passage of ore through the mixer was balanced with the rate of extrusion by the auger. The mixer, which has a shaft speed of 46 r.p.m., is driven by a 15-h.p., 760-r.p.m. motor.

The moisture content of the ore entering the auger is controlled at the mixer. Where the ore was too dry for successful extrusion, water was added as a fine spray on the mixer feed-belt ; where the ore was too wet, dry flue-dust or, less commonly, dried ground ore was introduced into the mixer trough by a vibrating feeder. Experience showed that this method could be relied upon to maintain a regular moisture content in the extruded pellets.

Auger

The machine is a standard 9-in. single-stage de-airing auger, the size denoting the internal diameter of the barrel (Fig. 3).

Power is transmitted from 50-h.p. motor (960 r.p.m.) and the main auger shaft revolves at 30 r.p.m.

The cast-iron body of the auger is in three sections :

(*a*) The lined feed-barrel, 2 ft. 9 in. long with a feed opening of 1 ft. 3 in. × 13 in., at the gearbox end, and terminating at the housing for the shredding device

(*b*) The de-airing chamber which is divided from the feed barrel by the shredding device

(*c*) The lined compression-barrel which continues from the de-airing chamber and is provided with a flange at the discharge end on which the extrusion die is mounted.

The liners in the feed barrel and compression barrel are keyed into the outer casing and their bores are splined, the splines running parallel to the horizontal axis of the machine to prevent the ore rotating.

The principal rotating parts are :

(i) The main auger shaft (30 r.p.m.) which extends through the feed barrel, de-airing chamber, and compression barrel

(ii) The shafts (40 r.p.m.) which carry the feed-packing blades.

In the lined feed-barrel the single-start scroll, 5 in. pitch, is so positioned that the discharge end is 1 in. clear of the shredding device to provide a pad of ore which acts as a seal. In the de-airing chamber there are seven knives on the shaft which, besides propelling ore forward, break up aggregates and so assist de-airing. The 5-in. pitch scrolls in the lined compression-barrel terminate with a 5¾-in. pitch, 2-start end worm. The purpose of this 2-start worm is to ensure a uniform forward movement over the whole area of the extrusion die. The feed-packing blades force the ore into the pitches of the scrolls.

The inlet side of the de-airing chamber is sealed by a shredding device which may take the form either of a perforated plate (Fig. 4) or a cone (Fig. 5). In addition to providing a seal, the purpose of this device is to shred the ore so that the maximum possible area is exposed to the de-airing action. The advantage of the shredder plate is that it provides a centre bearing for the auger shaft which would otherwise be unsupported over most of its length. On the other hand, when the webs of the plate become thin through

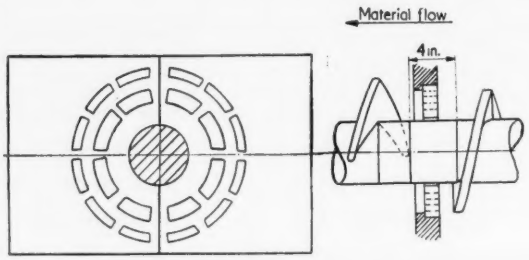


Fig. 4—Shredder plate

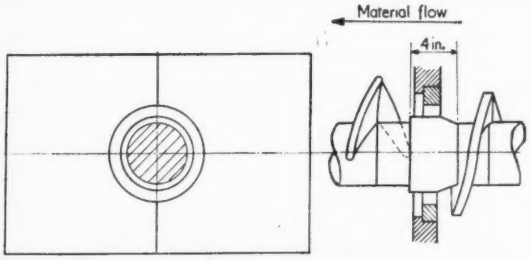


Fig. 5—Shredder cone

wear there is a danger of the centre being forced out and fouling the knives or scrolls in the de-airing chamber and compression barrel. With the cone shredder there is no such danger but, since the shaft is unsupported, its rolling action may prevent effective sealing of the de-airing chamber. Of a number of designs of shredder plates which were used that indicated in Fig. 4 is typical.

Dies

The closer the die can be placed to the end of the scroll the lower will be the power requirement for extrusion; at the same time there is a minimum distance between the die and the scroll below which the extrusion of the clay column is uneven. During the early experimental work the die shown in Fig. 6 was evolved. This die when fitted directly on to compression-barrel face gave erratic extrusion from the four holes but with a 2-in. thick distance piece the extrusions were even. The four 2-in. dia. holes were disposed, in this die, on a pitch circle of diameter 4 in., since previous experience had shown that with greater pitch circle diameters, such as 7 in., there was a tendency for the columns to turn in towards the axis of the die. A natural development of this practice was the provision of a die in which the ore column was guided positively towards the die holes, and the tapered die shown in Fig. 7 was evolved. This design was satisfactory and after some 1500 tons of pellets had been extruded the die holes had enlarged, by wear, to only $2\frac{3}{16}$ in. dia.

Cutting-Off Machine

A wire-type cutting-off machine was soon found to be unsatisfactory. The relatively coarse material caused the wire to drag through the moving columns instead of making a clean cut and, as a result, not only

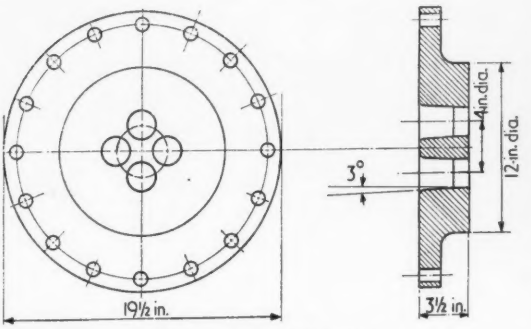


Fig. 6—Four-hole die

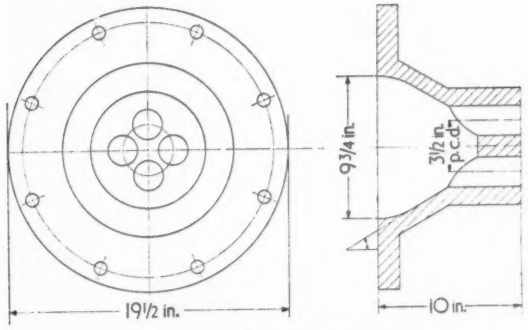


Fig. 7—Four-hole taper-nose die

was there an appreciable quantity of 'crumbs' from the cut pellets but the wire stretched and broke after a short life. A machine was designed in which a knife reciprocates across the flat, machined face of the die. The knife is 10 in. long and is made from 1-in. × $\frac{1}{8}$-in. spring steel, ground to cut on both the up- and down-strokes. The knife is carried by a frame which travels on two guide rods mounted in a spring-loaded main supporting structure (Fig. 8). The cutting force is applied by air at 70 lb./sq. in. operating a double-acting piston also mounted on the main structure. The stroke of the knife is 12 in. and the delay period between each stroke is regulated by a cam mechanism driven from the extrusion machine through a variable speed gear. The rate of cutting can be varied continuously between 20 and 180 cuts per minute and in operation this machine has been trouble-free. The knife is self-sharpening and the average life of a blade is about 400 tons of pellets.

Curing System

From the earliest experiments it was found that when the pellets are stored in batches immediately after extrusion—for example, in wire baskets of about 3 ft. cube—only those on the surface cure effectively in a reasonable time. Within the batch, free steaming is hindered and the natural toughening process is delayed or even suppressed. When a free circulation of air is provided the visible steaming is usually completed in about 20 min. and, after 40 to 45 min.,

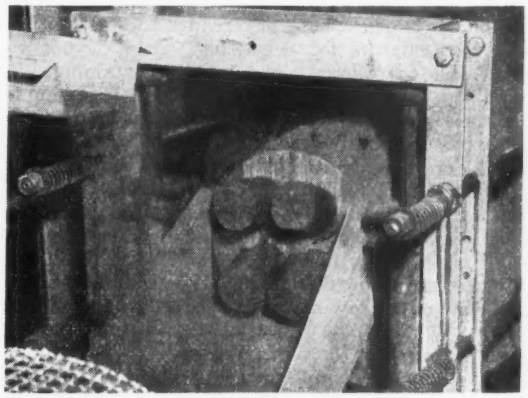


Fig. 8—Four-hole taper-nose die: 2-in. dia. pellets being cut off by blade of cutting-off machine on the upstroke

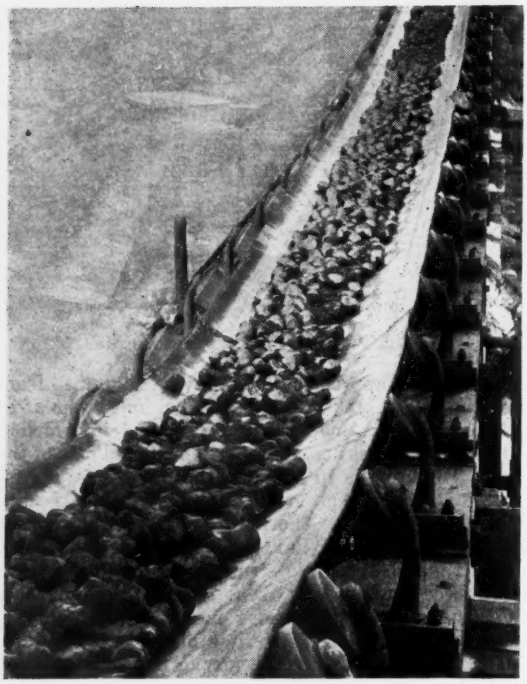


Fig. 9—Curing conveyor, before covering, from experimental plant to wagon-loading site

the pellets are tough and undamaged by handling under production conditions. The curing system for the experimental plant is a 27-in. wide rubber conveyor belt travelling at approximately 4 ft./min. over 170 ft. to a loading point (Fig. 9).

PELLETIZING

Materials

The available material was bedded ore which, after reclaiming, had been screened at $\frac{5}{8}$ in. for sintering. Since the uprising flue-dust—which is normally about 10% of the total ore weight—is returned to the Robins-Messiter beds and very largely becomes part of the ore fines, these fines contain substantial amounts of flue-dust which naturally reduce the plasticity of the raw mixture for pelletizing. As it is not yet advisable to pelletize ore mixtures containing $\frac{1}{2}$-in. and $\frac{5}{8}$-in. particles in regular and appreciable amounts, because of the likely damage to the auger, it is necessary to screen the ores at $\frac{3}{8}$ in. The moisture contents in the ore fines over more than half of the year lie in the range 14·1 to 17·0% (Table III).

In the screened ore, the + $\frac{1}{4}$-in. fraction varies from 3 to nearly 6% (Table IV). Generally, approximately half of these ore fines are below 72 mesh and one-quarter are below 200 mesh. The chemical analyses of some of the raw mixtures and pellets are given in Table V; these are typical of the Northamptonshire ore fines used at the Corby Works.

The ores are conditioned with flue-dust when too wet and, when the natural moisture content is below 16%, water is added before the mixing. Gas heating is available in the mixer and can be used for bringing the temperature of the material up to about 40° C. The aim of processing ores containing over 17% moisture has not yet been reached by the use of modifying agents such as burnt lime or commercial calcium hydroxide. Burnt lime, −36 mesh, was added to the extent of up to 3% and the hydrated lime up to 8%. In the former case, the effect on the temperature of the extruded column was very marked and temperatures as high as 94° C. were recorded. These additions reduced the plasticity of the ores and the pellets were too short or brittle.

Typical mixtures of Northamptonshire ores can have as much as 40% of flue-dust added and still give good self-hardening pellets without the addition of any binder. A mixture which is suitable for pelletizing has a feel which can be judged by a practised operator and this was soon found to be a more reliable guide than any physical test of extrudability.

Extrusion

As experience was gained it soon became possible to trace operating difficulties back to their origin. Successful auger operation depends on maintaining a constant rate of feed of properly conditioned material. While too high a feed results in the rapid choking of the feed opening, a low input of material not only results in a loss of vacuum but may also lead to an excessive wear on the scrolls immediately behind the shredder and the die. This is due to the face of the scrolls rubbing against compacted material instead of propelling it forward. For any type of raw material there is an optimum pitch of the scrolls. Too small a pitch prevents the full face of the scroll being effective in propelling the material forward, whilst too large a pitch incurs an excessive power consumption and increases the possibility of the material flowing back over the tips of the scrolls. With the stiff mixes extruded at Corby, there is no wear on the barrel liners, either at the feed end or in the compression barrel. Rather, the ore tends to adhere to the liners and to form a dense skin over which the moving material slips as it is carried forward by the scrolls. As the scrolls wear and become smaller in diameter the skin inside the liner becomes thicker so that, in effect, the barrel of the auger decreases in size during operation.

Table III

PERCENTAGE DISTRIBUTION OF DAILY MOISTURE CONTENT OF ORE FINES, 1952 and 1953

Moisture range, %			Percentage distribution 1952	1953
10·1	to	11·0	—	1
11·1		12·0	1	1
12·1		13·0	4	1
13·1		14·0	18	1
14·1		15·0	23	10
15·1		16·0	20	20
16·1		17·0	21	30
17·1		18·0	10	24
18·1		19·0	2	8
19·1		20·0	—	4
20·1		21·0	—	—
21·1		22·0	—	—
22·1		23·0	1	—
			100	100

Table IV

PARTICLE-SIZE DISTRIBUTIONS OF RAW MIXTURES—%, DRY BASIS

All values are those below the stated size

	Inches			B.S. Mesh										
	¼	$\frac{5}{16}$	⅛	7	10	14	18	25	36	52	72	100	150	200
1	94·3	87·3	76·6	71·7	67·5	64·0	61·2	57·5	54·3	49·5	41·8	35·2	26·6	23·1
2	96·2	90·8	82·2	77·5	72·9	69·0	65·9	62·1	58·8	54·6	46·8	39·7	30·7	26·5
3	95·6	90·5	81·6	77·3	73·0	69·5	66·5	63·0	59·8	55·6	48·3	41·9	33·3	29·3
4	97·0	93·0	85·0	80·6	76·5	72·7	69·6	65·8	62·6	58·1	50·6	44·0	35·2	31·7
5	94·7	88·0	78·5	73·9	69·8	66·2	63·0	59·5	56·7	52·8	45·9	39·7	31·7	28·3
6	95·9	90·7	81·7	77·7	74·1	71·1	68·7	65·8	63·2	59·3	53·4	47·9	39·5	36·2
7	95·5	88·8	78·0	73·3	69·0	65·4	63·0	59·8	57·2	53·7	47·7	42·0	34·1	31·0
8	95·9	90·9	81·9	77·2	72·7	68·8	65·8	61·7	58·6	53·8	46·6	39·9	31·0	27·5
9	95·7	89·3	80·8	76·1	71·7	67·7	64·9	61·1	58·0	53·7	47·1	41·3	33·3	30·0

The greatest wear of the scrolls takes place in the flight immediately behind the die and shredder and the wear decreases progressively in both sections towards the feed end. The result is that after a considerable tonnage of ore has been processed the scroll is tapered, instead of parallel. This appears to be in no way detrimental to the functioning of the machine and the quality of the pellets does not suffer, nor does the production rate greatly decrease, even when the scroll immediately behind the die has worn down to about 6 in. dia.

The fact that the scrolls wear at the periphery, rather than on the face, is an indication that the greatest amount of wear is caused by the ore sliding back between the scrolls and the barrel lining and may perhaps be considered as a measure of the efficiency of the auger as an extrusion machine. It does, however, have the advantage of allowing the scrolls to be built up on the periphery with hard, wear-resisting metal so that the life of a set of scrolls is prolonged. At those points where the frontal pressure is greatest, that is, at the die and the shredder, the face of the scroll is worn away, but the rate of wear of the face is still less than the rate of wear on the periphery. The indications are that the ores being processed were as large in the maximum particle size as was practicable, to the extent that some of the shredder plates holes become partly blocked, on occasion, by the relatively large ore pieces and sufficiently to cause at least a slight loss of vacuum.

When extruded without de-airing the pellets lack cohesion, for the strength of the vacuum-extruded pellet does not come from the compression received during extrusion but from the removal of the occluded

Table V

CHEMICAL ANALYSES OF RAW MIXTURES AND PELLETS—%, DRY BASIS

No.			Fe		SiO_2	Al_2O_3	CaO	S	C	Loss on ignition at 950° C.
			Total	Ferrous						
1	Raw Mixture		34·2	13·0	12·0	7·6	7·5	0·32	3·6	18·6
		Pellet	34·2	12·5	12·0	7·5	7·5	0·37	3·4	18·0
2	Raw Mixture		34·4	13·1	11·8	7·7	7·4	0·28	2·7	18·4
		Pellet	34·4	14·1	11·2	7·6	7·6	0·26	2·7	18·4
3	Raw Mixture		34·4	14·0	11·3	7·2	7·7	0·28	3·0	18·8
		Pellet	34·2	13·7	11·6	7·4	8·1	0·30	3·0	19·0
4	Raw Mixture		34·8	14·0	11·7	7·4	7·0	0·30	3·0	17·8
		Pellet	34·8	13·3	10·9	7·6	7·4	0·28	2·7	18·1
5	Raw Mixture		35·0	13·2	11·6	7·4	7·0	0·33	2·1	17·6
		Pellet	35·2	13·0	11·6	7·6	7·2	0·26	2·1	18·0
6	Raw Mixture		35·2	13·2	12·0	8·0	7·0	0·25	1·7	18·0
		Pellet	35·2	13·0	12·0	8·2	7·2	0·25	1·6	18·2
7	Raw Mixture		35·4	13·1	12·0	7·6	6·9	0·25	1·8	17·8
		Pellet	35·4	12·9	11·6	7·7	7·0	0·23	1·8	17·9
8	Raw Mixture		35·0	13·2	11·9	7·5	7·3	0·28	2·5	18·2
		Pellet	34·6	13·2	11·6	7·4	7·1	0·25	2·5	18·4
9	Raw Mixture		35·6	12·2	11·3	7·4	6·8	0·22	1·4	17·5
		Pellet	35·2	12·4	11·5	7·5	6·9	0·28	1·5	17·6

Table VI

PARTICLE-SIZE DISTRIBUTION OF PELLETS—%, DRY BASIS

All values are those below the stated size

No.	Inches			B.S. Mesh										
	¼	⅛	¼	7	10	14	18	25	36	52	72	100	150	200
1	98·7	96·2	90·4	87·1	83·7	81·1	78·8	75·9	73·2	69·4	63·8	57·8	49·1	45·3
2	98·6	95·7	89·8	86·6	83·3	80·6	78·2	75·4	73·1	69·3	63·2	57·6	48·6	44·9
3	98·9	96·5	90·3	87·2	84·0	81·2	79·0	76·2	73·7	69·2	61·6	54·9	46·9	43·7
4	99·0	96·5	90·1	86·2	82·7	79·8	77·3	74·2	71·7	67·4	60·7	54·5	45·8	42·6
5	98·8	95·8	88·3	84·8	81·5	78·8	76·9	73·9	71·5	68·1	62·3	57·1	48·4	45·6
6	98·7	96·4	89·2	85·6	82·2	79·4	77·0	74·3	71·8	68·6	63·0	57·8	49·3	45·7
7	99·4	97·7	92·4	89·0	85·4	82·2	79·8	76·7	74·1	70·7	64·5	58·5	49·3	44·5
8	99·0	96·7	90·0	86·7	83·2	80·1	77·6	74·7	72·3	68·7	63·3	57·6	48·0	44·7
9	98·6	95·5	88·4	84·4	80·8	77·7	75·3	72·6	70·1	67·0	61·3	56·1	47·6	43·5

air. Unless this is done effectively, the air remaining in the material after extrusion causes a swelling effect and there is no cohesion between the particles.

It is essential to maintain at least 25 in. Hg vacuum for the highest quality of pellets and none made with 22 in. Hg vacuum or less are likely to be satisfactory.

The experimental plant has an output rate of about 1·7 tons/hr. when extruding self-hardening pellets. The power consumptions per ton of pellets are, on average :

Mixer	2·9 kWh.
Auger, excluding vacuum pump	15·2 kWh.
Vacuum pump	0·8 kWh.

Curing

The best conditions for the production of self-hardening pellets are a moisture content of 16 to 16½% in the raw material and a temperature in the pellet of not less than 35° C. and, preferably, 50° C. Whilst temperatures in the pellets of up to 35° C. can result from friction in the auger it has been found that by applying gas heating in the mixer, the temperature of the pellets could be increased to 50° C. The self-hardening effect is not solely a result of a moisture loss since, for the most part, the pellets lose only about ½% unit of moisture over the 45-min. curing time.

The normal cylinder is seemingly the most suitable shape of pellet. If short pellets are made in which the length is only, say, half the diameter, breakages during handling are inevitable. In the normal cylinder the area of the ends is one-third of the total surface area and it is through these rough ends, rather than through the more compact body of the cylinder, that much of the steaming takes place. If the length of the pellet is allowed to increase until it is double the pellet diameter, there is a noticeable tendency for such pellets to have their surfaces slightly cracked. These cracks can often be seen to originate and spread and later join with others to form fissures which are possible points of weakness. In the curing process, there may be some redistribution of the moisture content of the pellet and although curing involves no great loss of moisture it cannot be brought about by chilling the pellets by cold air. Pellets so treated are weak and it would seem that a fundamental of the curing process is the expiration of water from capillary passages until these are so reduced in size that a cohesive force operates and imparts toughness and strength to the pellet.

Properties of the Pellets

Particle-Size Analysis—The examples of the particle-size distributions given in Table VI are typical : they correspond to the gradings of the ore fines in Table IV. It will be seen that in the pellets the +¼-in. content was of the order of ½ to 1½% and that roughly half the pellet weight is in the small particles −150-mesh fraction. Many examples have been recorded of size distributions of the form shown in Fig. 10 ; the shape of the distribution curve is characteristic.

Density and Porosity—The ore fines, before extrusion, have a bulk density of 100 to 104 lb./cu. ft. As extruded, the column of ore has an apparent density of 140 lb./cu. ft. The cured pellets, as charged to the furnace, have a bulk density of 87 lb./cu. ft. The apparent densities of pellets, on both the wet and dry bases, extruded from normal ore mixtures are closely related to their moisture content (Fig. 11), and the porosity values of the dried pellets are closely related to the original moisture contents (Fig. 12). The relationship between percentage porosity (P) and percentage moisture (M) is :

$$P = 20{\cdot}5 + 0{\cdot}98\,M$$

In the lump ores used at Corby the porosities are usually between 25 and 35% and, as the pellets would be charged to the blast-furnaces with a moisture content of some 15%, the pellet porosities of about 35%

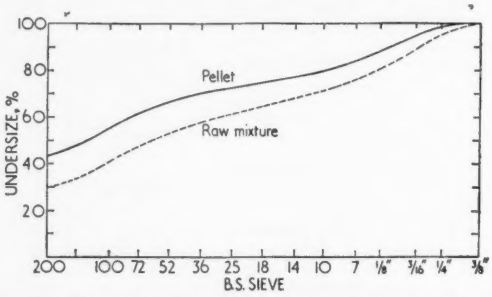


Fig. 10—Typical particle-size distributions of raw mixtures, before extrusion, and of pellets

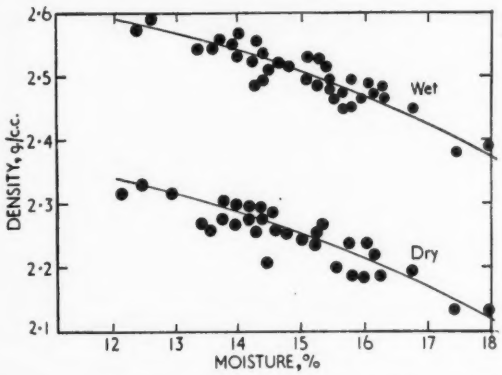


Fig. 11—Effect of moisture content of pellets on pellet densities, wet and dry bases

to be expected would compare favourably with the lump ores.

Compressive Strength—The relationship between compressive strength and moisture content of pellets is shown in Fig. 13 in which each point is the average of ten determinations, themselves closely grouped around the average value. While there may be a general impression of rather low compressive strengths, the pellets can withstand handling and storage under production conditions.

Shatter Test—The resistance to breakage is conveniently measured in the apparatus required by the British Standard Shatter Test for Coke. The coke test has been modified for testing pellets :

(*a*) By increasing the severity from four drops to eight drops

(*b*) By expressing the number of pellets surviving the test without breakage as a percentage of the original 25 pellets tested.

The aim has been to produce pellets which will survive eight 6-ft. drops without deterioration. Figure 14 shows the effect of moisture content on the resistance of pellets to the shatter test and also the powerful effect of the number of drops specified for the test. If the shatter test of eight drops is the best

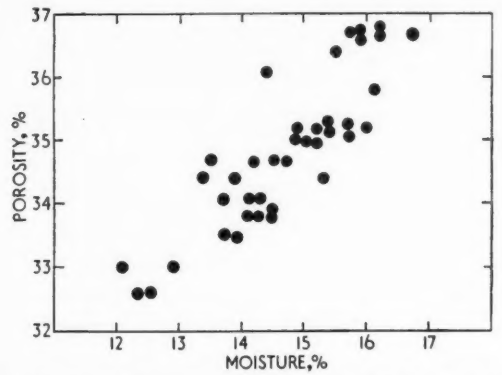


Fig. 12—Effect of moisture content of pellets on pellet porosity

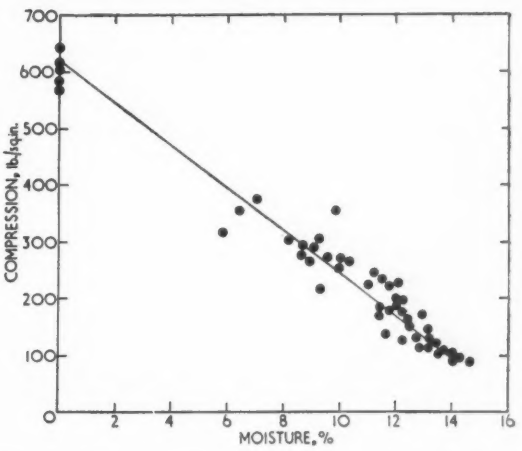


Fig. 13—Effect of moisture content of pellets on pellet compressive strength

criterion of strength for the handling conditions at Corby, the indications are that a moisture content of about 14% in the pellets would be the minimum permissible.

BLAST-FURNACE TRIAL

Some 1400 tons of pellets were made in the experimental plant over 110 shifts at the average rate of 1·7 tons per net operating hour, and were charged into No. 1 blast-furnace over five days in March, 1952. As there were no means of reducing the disparity between the rates of production and consumption of the pellets the trial was, of necessity, limited in scope.

The furnace burdens for the five-day trial period and for the corresponding periods before and after are summarized in Table VII.

In the lump ore, at the time of the trial, the +2-in. content ranged from 25 to 36%. The apparent $-\frac{1}{4}$-in. content, *i.e.*, the fraction obtained by screening the ore in the as-used state, varied from 2 to 8% but the true content was as high as 23%. In the sinter the +2-in. content, at this time, varied from nil to 12% and the $-\frac{1}{4}$-in. content varied from 7 to 30%. The results of the routine shatter tests taken at

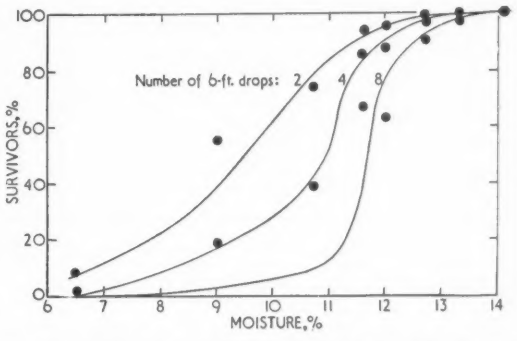


Fig. 14—Effect of moisture content of pellets on percentage surviving two, four, and eight 6-ft. drops

Table VII

BLAST-FURNACE PRACTICE

	Before Trial, 7 days	Trial, 5 days	After Trial, 5 days
Burden, lb./ton iron			
Lump Ore	3,251	3,044	3,650
Pellets	—	1,044	—
Sinter	1,814	1,209	1,525
Fluxes and scrap	741	809	800
Total, lb./ton iron	5,806	6,106	5,975
Coke, lb./ton iron	1,712	1,794	1,747
Slag, lb./ton iron	1,540	1,534	1,550
Iron, tons/day	565	535	547
tons/day/sq. ft. hearth	1·80	1·70	1·74
tons/day/100 cu. ft. working volume	2·08	1·97	2·01
Coke, tons/day	432	428	427
tons/day/sq. ft. hearth	1·38	1·36	1·36
Flue-dust, lb./ton iron	200	290	278
Iron Analysis, %			
Si	0·93	0·96	1·02
Mn	0·52	0·57	0·60
S	0·31	0·29	0·28
P	2·06	2·10	2·13
Slag Analysis, %			
SiO_2	34·0	33·9	34·5
Al_2O_3	23·4	23·0	22·9
CaO	32·4	33·1	32·3
MgO	6·7	6·9	6·8
MnO	1·3	1·4	1·3
S	1·5	1·5	1·4
FeO	1·1	0·8	1·1

this time, which are given in Table VIII, have been grouped in order of merit for the eight-drop test. All the pellets represented by these test data were free-running in the high-line bins and of good appearance in the scale car, and in the blast-furnace skip (Fig. 15).

The practice at the time of the pellet trial was notable for the high driving-rate of the furnaces. Over the previous year the average daily hot-metal production from No. 1 furnace, less the pig iron equivalent of all the scrap in the burden, was 390 tons per operating day. This output, on the same basis, the week before the trial with the pellets, rose to the record value of 456 tons/day. During the trial the daily make averaged 425 tons/day and afterwards it rose slightly to 437 tons/day (Fig. 16).

The hot-metal analyses plotted in Fig. 17 indicate that the three periods are indistinguishable in this respect. Over the three periods the blast-furnace drove smoothly at a blowing rate of 40,000 to 42,000 cu. ft./ min.

Table VIII

SHATTER TEST ON PELLETS SAMPLED FROM SCALE CAR: PERCENTAGE SURVIVORS

Test No.	Number of 6-ft. drops				
	4	8	16	32	40
1	100	100	88	28	4
2	96	92	92	68	56
3	96	92	80	44	28
4	96	88	88	28	24
5	92	84	60	12	0
6	88	88	56	8	4
7	80	72	44	16	0

Fig. 15—Pellets in blast-furnace skip

This brief experience of pellets in the blast-furnace burden was encouraging and was considered to support the case for a pelletizing plant of larger capacity.

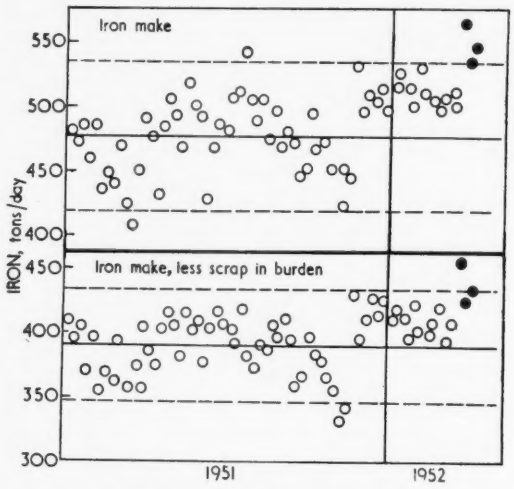
PILOT PLANT

If all the uprising ore fines were pelletized, the usage in the blast-furnaces at Corby would be approximately the same tonnage as the hot-metal production from burdens which included only moderate amounts of scrap. The general objectives of the pilot plant are to provide such a supply to one of the blast-furnaces and, if possible, to process the $-\frac{5}{8}$-in. fines without drying and grinding or the use of binders and without firing the pellets.

No ore-drying equipment has been installed in the pilot plant but, as the possibility of extruding ores as coarse as $\frac{5}{8}$ in. is yet uncertain, a pan mill has been included in the flowsheet (Fig. 2).

Layout

The plant was installed in an existing building housing two identical Dwight Lloyd sinter strands



Points are daily averages over weekly periods; the three solid circles represent the weeks before, during, and after the blast-furnace trial

Fig. 16—Daily iron production of No. 1 blast-furnace,

which were part of the original Corby ore-preparation equipment built in 1934. The sinter strands were fed from a common set of storage bins and conveyor belts, which were retained. One strand was dismantled to accommodate the pelletizing plant. The building, which has a ground area of 119 ft. × 61 ft., had the advantage of existing floor ways and of structural steelwork of sufficient strength to accommodate the pelletizing plant (Fig. 18). The original sinter-discharge system, the skip hoist-way and the sinter storage bin have been retained.

Towards the end of last year the plant was sufficiently forward for preliminary trials although some ancillaries have not yet been completed (February, 1954).

Plant

The mixture for pelletizing is supplied from the storage bins and passes into the pan mill which is situated on ground level. The mill is of the solid-bottom grid-discharge type and has two rolls which weigh 6½ tons each and is driven by a 50-h.p. motor. The mill rotates at 16·5 r.p.m. giving a discharge capacity of about 25 tons/hr. The milled ore then passes to a surge hopper, on the third floor, of 6-tons capacity from which a regulated feed supplies the 9-ft. double-shaft mixer on the second floor. The extrusion auger has been placed at the opposite end of the building from the mixer to allow for ancillary equipment (such as a rounding-off drum for the extruded pellets) should this be necessary.

The extrusion auger is a two-stage machine incorporating a mixer section immediately preceding the scroll which feeds the shredding device. The machine is powered by a 175-h.p. motor and the top and bottom shafts rotate at 34 and 28 r.p.m., respectively. The inside diameter of the compression barrel is 23½ in. and liners of various sizes, with corresponding scrolls, may be fitted. In the preliminary trials the machine has been operated as a 19-in. auger ; 10-in. and 14-in. pitch scrolls have been used in the compression barrel with one- and two-start end worms. These alternative settings, together with the use of spacer rings behind the die, up to 6 in. in thickness, have been the bases for a series of preliminary tests. Flat dies have been used for extruding 2-in. and 2½-in. dia. pellets and the die shown in Fig. 19, which has

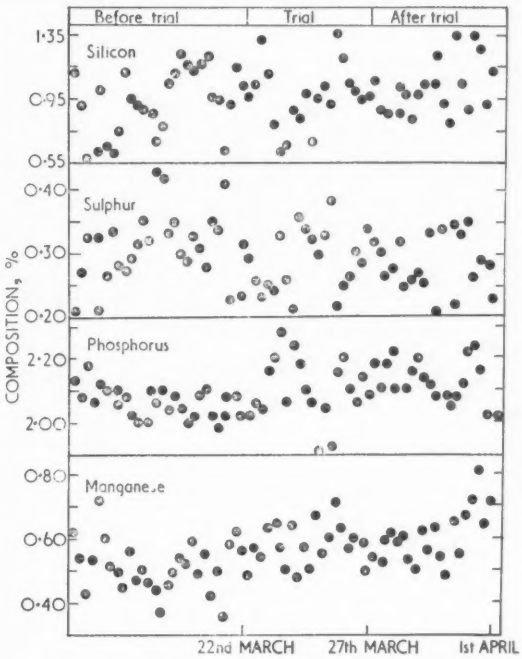


Fig. 17—Hot-metal analyses, No. 1 furnace

thirty-six 2-in. dia. holes, has been satisfactory in in service.

The cutting-off machine (Fig. 19) is air-operated and the cutting knife, which is 22 in. long, has a horizontal stroke of 24 in. After cutting, the pellets pass through a mechanical spreader to the curing conveyors shown in Fig. 20. These trays, which are 9 ft. wide, are of mild steel, galvanized, and are mounted on link conveyor-chains driven through variable-speed gearing. The normal curing time is 45 min. after which the pellets are collected in a hopper which discharges at regular intervals into the skip. This operation is controlled by a limit switch actuated by trip levers attached to the final run of the curing trays. From the top of the skip-way the pellets travel down a chute and fall vertically 20 ft. into the main storage bunker.

The pan-mill feed varies between 7 and 11½% in the +¼-in. fraction while the product contains 5½ to 9½% + ¼-in. Approximately half of the product is −72-B.S. mesh and one-third −200-B.S. mesh. Although this ore contains a greater proportion of +¼-in. material than that processed in the experimental unit, it does not appear to have had any detrimental effect on the product.

In the preliminary trials the plant has produced pellets at the rate of 12 to 15 tons/hr. at an overall power consumption of 15 kWh. per ton of pellets.

Table IX

SIZE GRADINGS, %

	Calcines, −⅜ in.	Calcines, −⅛ in.	Sierra Leone Concentrates	Kiruna *KC2* Concentrates
− ⅜ in. to + ¼ in.	1·2	nil	nil	nil
− ¼ in. to + 5/16 in.	1·9	nil	nil	5·2
− 5/16 in. to + ⅛ in.	4·0	0·6	0·7	17·8
− ⅛ in. to + 7 mesh	5·0	2·9	0·9	9·0
− 7 mesh to + 10 ,,	4·3	1·7	1·4	8·1
− 10 ,, to + 14 ,,	3·3	2·3	1·3	6·9
− 14 ,, to + 18 ,,	3·8	1·7	1·0	5·5
− 18 ,, to + 25 ,,	5·3	2·3	2·6	6·3
− 25 ,, to + 36 ,,	3·5	2·3	5·9	3·9
− 36 ,, to + 52 ,,	5·0	3·5	14·8	4·3
− 52 ,, to + 72 ,,	9·4	5·7	24·0	5·4
− 72 ,, to + 100 ,,	8·7	5·7	18·5	3·7
−100 ,, to + 150 ,,	14·0	13·2	18·9	6·7
−150 ,, to + 200 ,,	4·7	5·2	5·9	4·8
−200 ,,	25·9	52·9	4·1	12·4

ENRICHED PELLETS

It has to be accepted for the present that the concentration, by gangue removal, of the ore mixtures used at Corby is not practicable in any known process.

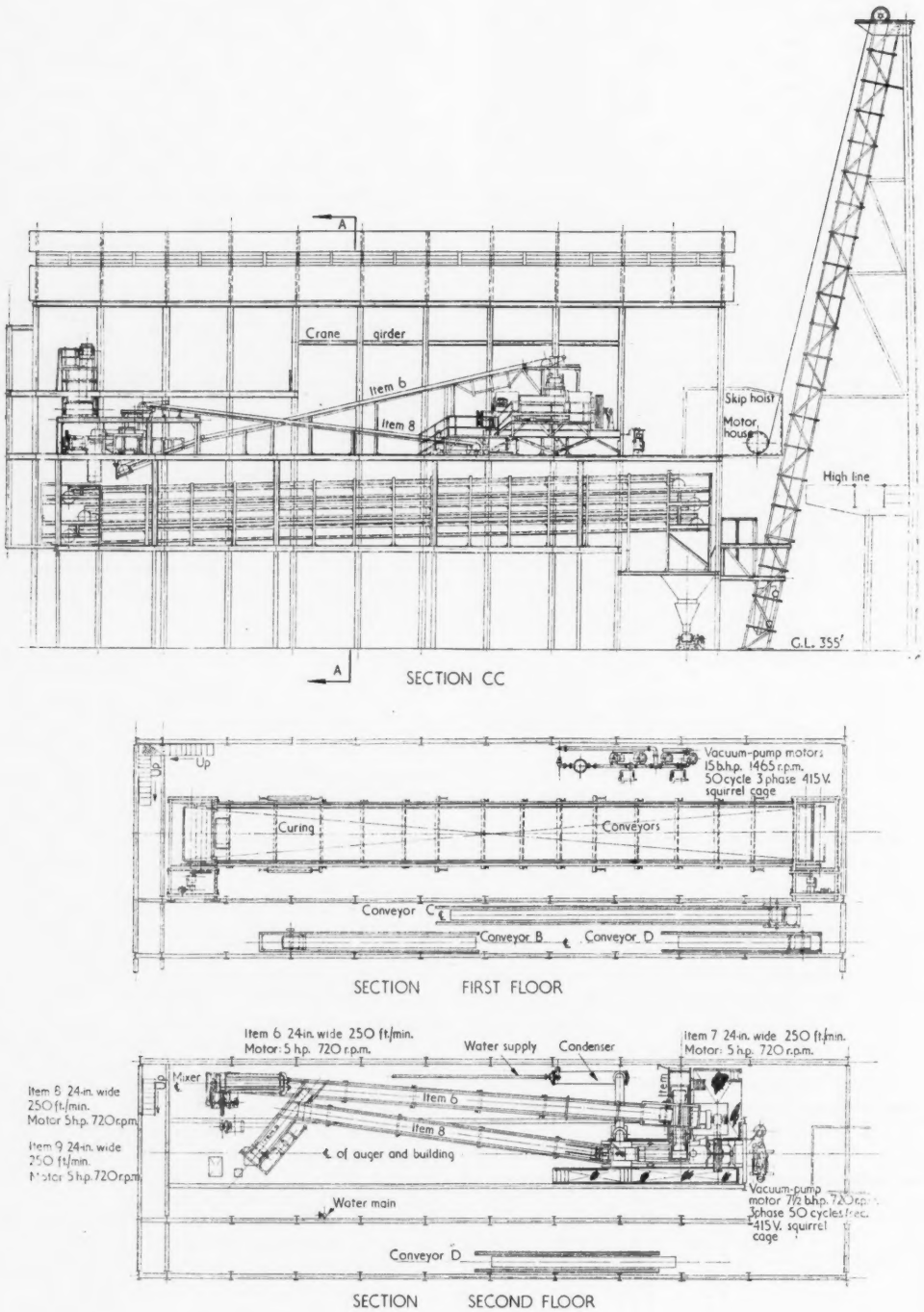
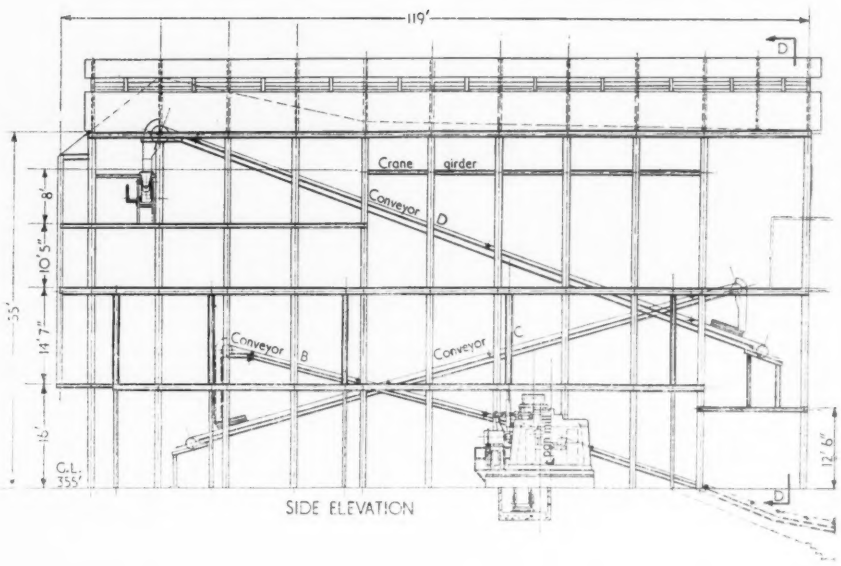


Fig. 18—Layout of pilot pelletizing plant



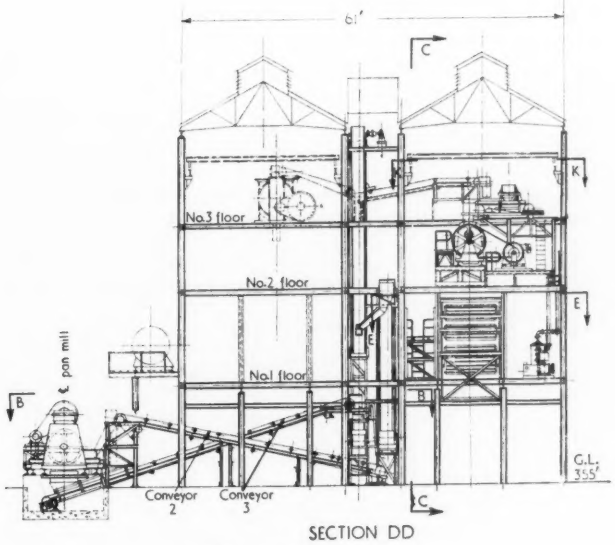


Fig. 18—(*Continued*)

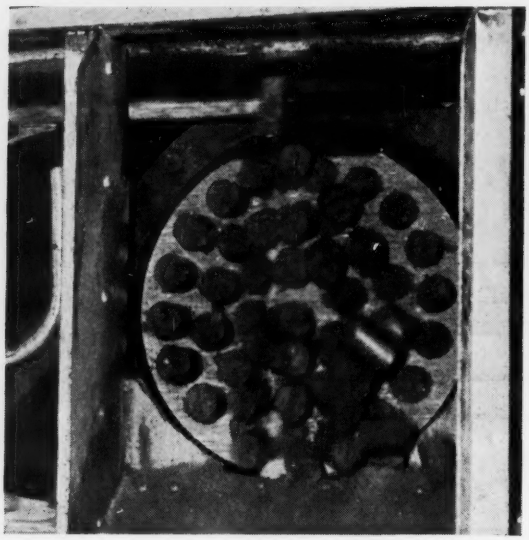


Fig. 19—Die and cutting-off machine

An increase in iron content can, of course, be achieved by the removal of the volatile content.

In pelletizing, enrichment is also possible by the addition of higher grade ores to the local material and a further concentration can be obtained by firing the enriched pellets.

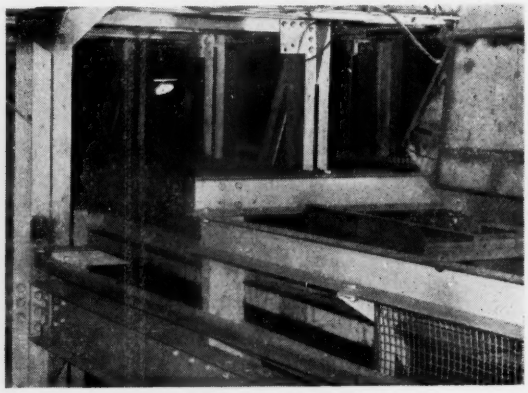


Fig. 20—Curing system

Work in the experimental unit has shown that up to 40% of added materials can be incorporated into pellets based on the run-of-plant ore fines (Table IV) and the enrichers so far used have included Sierra Leone concentrates, Kiruna *KC*2 concentrates, and the fines recovered after the calcination of Northamptonshire lump-ore mixtures. The calcines were screened at $\frac{3}{8}$ in. and $\frac{1}{4}$ in. but the imported ores were used without any preliminary treatment. The size gradings of the added materials are given in Table IX.

Materials

The pellet firing experiments cover the following materials :

Table X

PHYSICAL PROPERTIES OF PELLETS AND SIZE GRADING OF MIXTURES BEFORE EXTRUSION

Material		(i)		(ii)		(iii)		(iv)		(v)		(vi)*	
Pellet													
Diameter, in.		2		2		2		2		2		2	
Length, in.		$1\frac{3}{4}$		2		$1\frac{3}{4}$		$1\frac{3}{4}$		$1\frac{3}{4}$		$1\frac{3}{4}$	
Apparent Density, g./c.c.		2·13		2·22		2·15		2·25		2·59		1·92	
True Density, g./c.c.		3·26		3·36		3·22		3·40		3·71		2·93	
Porosity, %		34·6		33·9		33·2		33·8		30·2		34·4	
Size grading, %		Mixture for extrusion	Pellet	Mixture for extrusion	Pellet	Mixture for extrusion	Pellet	Mixture for extrusion	Pellet	Mixture for extrusion	Pellet	Mixture for extrusion	Pellet
Minus	Plus												
$\frac{3}{8}$	$\frac{1}{4}$	6·9	1·3	4·3	1·4	9·5	1·8	5·4	1·3	1·5	1·2	5·4	1·3
$\frac{1}{2}$	$\frac{3}{16}$	9·8	4·9	6·4	3·1	4·4	2·7	7·5	3·0	2·8	2·7	7·9	4·0
$\frac{3}{16}$	$\frac{1}{8}$	11·9	8·0	8·5	7·1	7·3	3·6	11·1	6·6	4·5	5·0	9·5	7·7
$\frac{1}{8}$	7	5·9	4·1	4·7	4·0	3·2	3·0	5·3	2·9	6·1	4·6	4·2	4·3
7	10	5·2	3·2	4·4	3·6	3·3	2·9	4·4	6·0	6·7	4·5	3·6	3·9
10	14	4·0	3·3	4·0	3·1	3·3	3·2	3·5	2·7	7·0	4·9	3·3	3·3
14	18	2·8	2·5	2·8	2·4	3·0	2·8	2·9	2·7	5·1	4·7	2·8	2·7
18	25	3·2	2·8	3·8	2·7	3·8	4·3	3·7	3·3	6·5	3·0	3·6	3·2
25	36	2·3	2·3	3·1	2·5	3·8	3·8	2·9	2·6	4·2	2·8	2·9	2·3
36	52	2·4	2·9	4·3	3·1	5·0	3·5	3·1	0·7	5·2	3·8	1·9	2·1
52	72	6·5	4·5	6·6	5·7	7·1	7·6	7·7	8·5	6·5	5·7	7·8	6·6
72	100	4·5	4·4	5·8	5·2	4·9	5·6	6·2	6·1	5·3	5·3	5·4	5·3
100	150	5·5	3·7	8·0	8·5	6·5	8·0	6·2	7·2	7·6	7·7	6·6	8·1
150	200	1·5	1·6	3·3	4·1	3·0	3·0	3·2	3·3	3·5	3·3	3·7	3·9
200		27·6	50·5	30·0	43·5	31·9	44·2	26·9	43·1	27·5	40·8	31·4	41·3

* Mixture (ii) with 8% of coal.

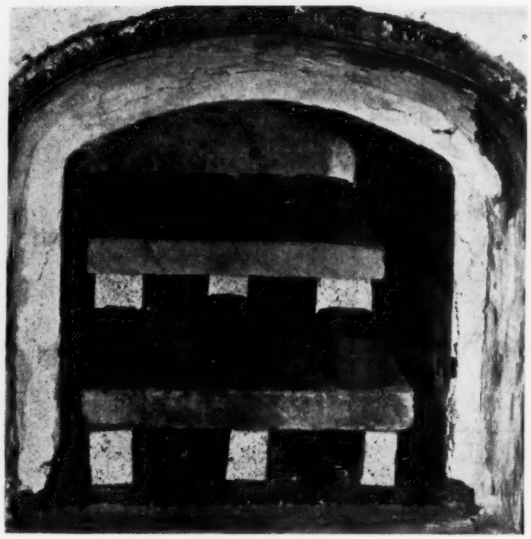


Fig. 21—Gas-fired muffle furnace with charge of pellets, before firing

(i) —$\frac{3}{8}$-in. bedded ore-fines, without flue-dust
(ii) —$\frac{3}{8}$-in. bedded ore-fines containing 10% of flue-dust
(iii) As (ii), containing $6\frac{1}{2}$% of limestone
(iv) As (ii), containing 10% of Kiruna *KC*2 concentrates
(v) As (ii), containing 40% of Kiruna *KC*2 concentrates.

Mixture (ii), is, of course, the type of material pelletized in the experimental and pilot plants. While it was necessary to determine the properties of fired pellets made from the ore mixtures without flue-dust [(i)] and from a mixture containing both flue-dust and limestone [(iii)], the results were so similar to those obtained from the mixture containing flue-dust [(ii)] that it should suffice, in the present account, to report only these. Again, only the results of the firing experiments with the material containing 40% Kiruna *KC*2 concentrates [(v)] need be reported ; this mixture is, of course, of interest as the present practical limit of incorporation of an added material to the Corby ore-fines. The principal physical properties of the pellets made with these materials and the size gradings of the mixtures before extrusion are given in Table X.

Table XII

DENSITIES AND POROSITIES OF NATURAL PELLETS AFTER FIRING

Property	Soaking Time, min.	Firing Temperature, ° C.			
		1150	1175	1200	1250
Apparent Density, g./c.c.	15	2·06	2·13	2·24	2·55
	30	2·05	2·06	2·21	2·58
	45	2·07	2·09	2·19	2·65
	60	2·31	2·12	2·26	2·63
True Density, g./c.c.	15	3·85	3·82	3·89	3·98
	30	3·84	3·76	3·97	3·99
	45	3·89	3·86	4·04	4·03
	60	3·99	3·90	3·95	4·05
Porosity, %	15	46·2	44·2	42·4	35·9
	30	46·6	45·2	45·2	35·3
	45	46·8	45·8	45·8	34·2
	60	42·1	45·6	42·7	35·0

In the firing treatments temperatures approaching the fusion point have to be used otherwise the pellets are extremely brittle and friable and, in addition, an oxidizing atmosphere is necessary to effect sulphur removal.

Firing Cycle

For each test, some 60 pellets, previously dried at 105° C. for 24 hr., were placed on stools arranged in two tiers in a gas-fired muffle furnace (Fig. 21). The furnace was heated at the overall rate of 5° C./min. up to the firing temperature : this rate was the maximum for the furnace. An upper limit to the firing temperature is imposed by the rapid softening and collapse of the pellets, which would occur at temperatures above 1250° C. After holding at the firing temperature for the required time, the furnace was allowed to cool about 200° C. before the pellets were removed.

Natural Pellets

Pellets made from the mixture (ii), above, are conveniently described as *natural pellets*. The iron and sulphur contents of these after firing are given in Table XI and the density and porosity values in Table XII. It will be seen from Fig. 22*a* that there is a progressive decrease in the sulphur content of the fired pellets to a minimum of 0·02% as the firing temperature is increased to 1250° C. The number of pellets surviving the shatter test increased as the firing temperature was raised and, with two exceptions, at any

Table XI

CHEMICAL ANALYSES OF NATURAL PELLETS AFTER FIRING—%

Firing Temp., °C.	1150				1175				1200				1250			
Soaking Time, min.	15	30	45	60	15	30	45	60	15	30	45	60	15	30	45	60
Total Fe	46·2	46·7	45·4	45·6	45·9	45·3	45·6	45·8	45·7	45·3	45·3	46·2	46·2	46·2	45·4	46·4
Ferrous Fe	9·2	11·8	5·7	11·2	5·7	5·7	5·6	7·8	5·1	5·1	5·1	8·9	5·8	4·5	3·1	9·4
Total S	0·26	0·25	0·23	0·30	0·23	0·22	0·16	0·20	0·19	0·13	0·10	0·10	0·10	0·02	0·02	0·02
S present as sulphate	0·17	0·12	0·17	0·18	0·18	0·16	0·13	0·12	0·15	0·09	0·06	0·06	0·03	0·02	0·01	0·02

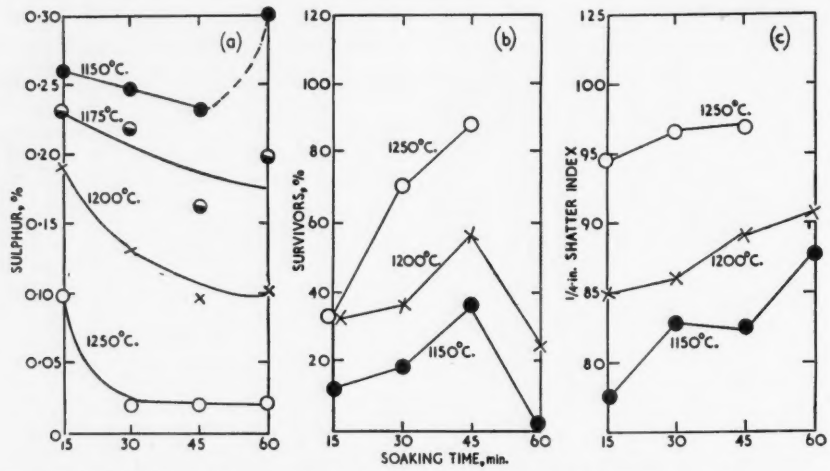


Fig. 22—Effect of firing temperatures and soaking times on (*a*) sulphur content of fired natural pellets, (*b*) percentage survivors after eight 6-ft. drops (natural pellets), (*c*) percentage + ¼ in. after eight 6-ft. drops (natural pellets)

temperature level, the percentage of survivors increased as the soaking time was lengthened (Fig. 22*b*). Pellets soaked for 15 min. at 1250° C. were no stronger. as regards the number of survivors, than those soaked for 15 min. at 1200° C., but comparison of the shatter indices (Fig. 22*c*) clearly distinguishes the effect of the various firing treatments.

The true densities of the fired pellets were some 15 to 20% higher than those of the dried, unfired pellets, but the apparent densities of the fired pellets ranged from nearly 10% less to about 20% more than those of pellets in the unfired state (Fig. 23). The sudden increase in these latter relative values when the firing temperature was raised caused a marked decrease in the relative porosity values (Fig. 24).

The compressive strength was found to increase as the firing temperature was raised and with the longer soaking times at each of the firing temperatures. Values between 1300 and 1500 lb./sq. in were obtained after firing at 1250° C.

Northamptonshire–Kiruna pellets

The ore mixture (ii) when enriched by the addition of 40% Kiruna *KC*2 concentrates has an iron content of about 47½% and a sulphur content of approximately half that of the mixture of the Northamptonshire ores with flue-dust. The extent to which enrichment by devolatilization is possible is, of course, limited since these pellets in the dried state have a loss on ignition at 950° C. of less than 11%.

There was a progressive reduction in sulphur content as the firing temperature was increased and at 1250° C. the sulphur was completely removed (Fig. 25*a* and Table XIII). Firing at 1200° C. and 1250° C. for longer than 15 min. gave a complete survival of the

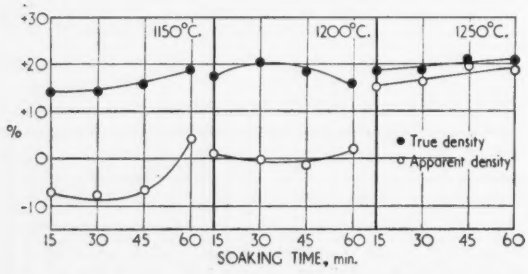


Fig. 23—Effect of firing temperatures and soaking times on the relationship between true and apparent densities of fired pellets and those of air-dried pellets (natural pellets)

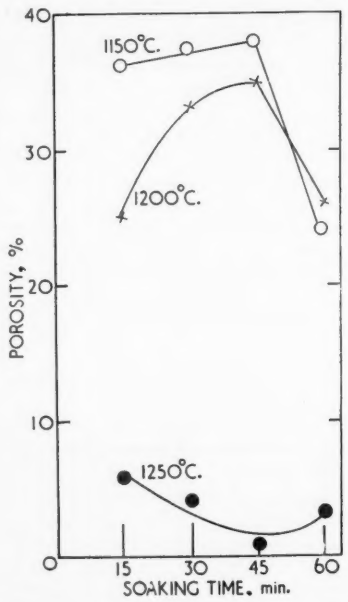


Fig. 24—Effect of firing temperatures and soaking times on the porosity of fired pellets relative to air-dried pellets (natural pellets)

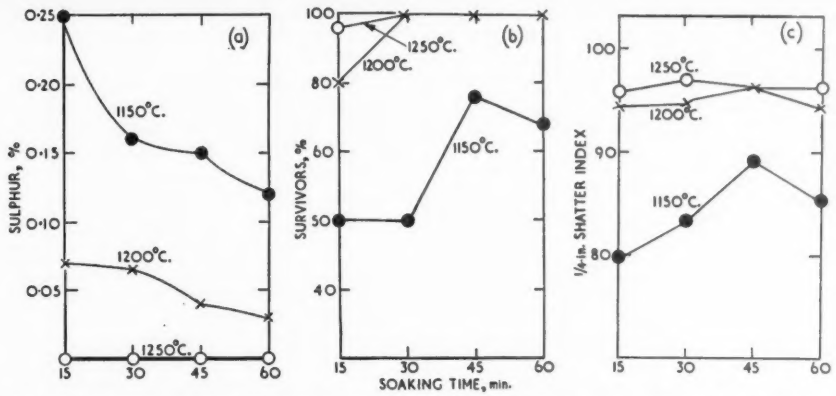


Fig. 25—Northamptonshire–Kiruna pellets : effect of firing temperatures and soaking times on (*a*) sulphur content of fired pellets, (*b*) percentage survivors after eight 6-ft. drops, (*c*) percentage + ¼ in. after eight 6-ft. drops

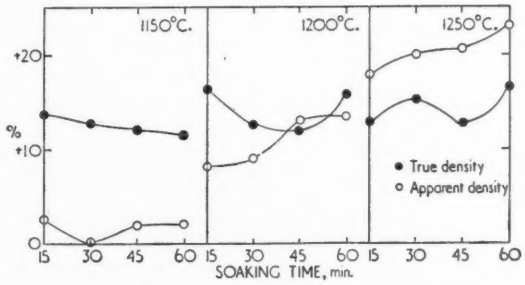


Fig. 26—Northamptonshire–Kiruna pellets: effect of firing temperatures and soaking times on the relationship between true and apparent densities of fired pellets and those of air-dried pellets

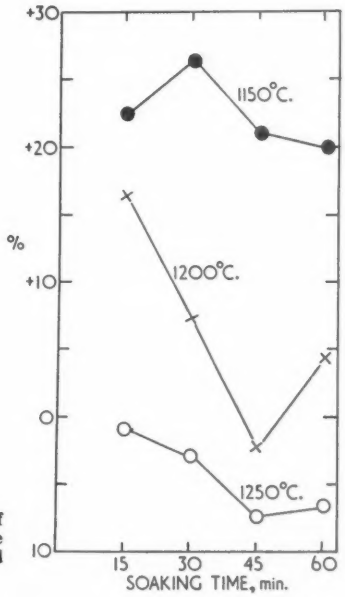


Fig. 27—Northamptonshire–Kiruna pellets: effect of firing temperatures and soaking times on the relationship between the porosities of unfired and air-dried pellets

Table XIII

CHEMICAL ANALYSES OF NORTHAMPTONSHIRE–KIRUNA PELLETS AFTER FIRING

Firing Temp., ° C.	1150				1200				1250			
Soaking Time, min.	15	30	45	60	15	30	45	60	15	30	45	60
Total Fe	54·0	53·2	53·8	53·2	53·6	53·6	52·6	52·8	53·0	53·4	53·0	52·6
Ferrous Fe	3·7	1·6	2·7	1·3	7·0	1·2	0·7	0·7	0·7	0·9	0·7	0·9
Total S	0·25	0·16	0·15	0·12	0·07	0·06	0·04	0·03	nil	nil	nil	nil
S present as sulphate	0·24	0·16	0·15	0·11	0·03	0·05	0·04	0·03	nil	nil	nil	nil

Table XIV

DENSITIES AND POROSITIES OF NORTHAMPTONSHIRE–KIRUNA PELLETS AFTER FIRING

Property	Soaking Time, min.	Firing Temperature, °C.		
		1150	1200	1250
Apparent Density, g./c.c.	15	2·66	2·80	3·06
	30	2·59	2·82	3·11
	45	2·64	2·93	3·13
	60	2·63	2·94	3·19
True Density, g./c.c.	15	4·22	4·32	4·19
	30	4·19	4·18	4·28
	45	4·16	4·15	4·19
	60	4·14	4·30	4·33
Porosity, %	15	37·0	35·2	29·8
	30	38·2	32·5	27·4
	45	36·6	29·4	25·0
	60	36·4	31·6	26·2

eight 6-ft. drop shatter test (Fig. 25*b*) and in all cases the ¼-in. shatter index was about 95% (Fig. 25*c*).

The true and apparent densities and the corresponding porosity values are given in Table XIV and these data, relative to the values for the dried, unfired pellet (Table X) are shown in Figs. 26 (densities) and 27 (porosities).

Conclusions from Experimental Work

It can be concluded from this experimental work that the admissible firing temperatures lie within a very narrow range the upper limit of which is set by rapid softening and collapse and it is to be expected that any furnace for firing pellets under production conditions would require to be very closely controlled. The possibility arises of incorporating the fuel for the firing treatment in the pellets themselves and pellets have been made containing both low-grade coal and coke breeze. In laboratory firing experiments on pellets made from mixture (ii) with 8% of coal (*see* mixture (vi), Table X) the indications are again that it is necessary to fire at temperatures very near to the fusion point which in this case is only about 1100° C.

ASSESSMENT OF DEVELOPMENTS

Although the possibilities of the vacuum-extrusion process for the production of self-hardening pellets have not yet been fully explored at Corby, some assessment of the developments can be made.

The process is the one most suited to the agglomeration of the uprising materials where only a small degree of pre-preparation—if any—is admissible and when an unfired agglomerate is to be used in the blast-furnaces.

The empirical approach of experimenting with the raw materials and the type of plant to be used under production conditions has advantages which outweigh the disadvantages. The drawbacks are that the search for the best materials and design of plant may be relatively more expensive and the time taken to make changes will certainly be greater than they would be on small-scale models but, on the other hand, the data gained require no conversion from the small to the full scale.

That much of the necessary plant can be obtained as standard equipment is an advantage. In the Corby plants, the cutting-off machines only were specially designed ; in the pilot plant the pan mill, the double-shaft mixer, and the curing system have been used without modification and have given little trouble in operation. The extrusion auger has been adapted to operate both as a 19-in. and as a 16-in. machine but neither, as yet, for sufficiently long to provide reliable technical and economic data in the pelletizing plant or in the blast-furnaces.

The process does not require more than ordinarily experienced and conscientious operators.

The production costs of unfired pellets are likely to be attractive but the additional charges for firing may well be greater than is worthwhile.

The immediate aim at Corby is to have further experience of unfired pellets in a blast-furnace and, moreover, to use this method of agglomeration, as circumstances allow, for the enrichment, the fluxing, and even the fuelling of that part of the burden which is supplied by the ore fines.

Acknowledgments

The author has to thank the Directors of Stewarts and Lloyds Ltd., for their permission to present this paper and for the support and encouragement which the work has been given from the beginning. The Management of the Iron and Steel Works at Corby have afforded every facility for using extruded pellets as blast-furnace burden and have borne the vicissitudes of experimentation with a kindly forbearance. The New Development Section of the Company has provided engineering services for the projects, and the Members of the Blast-furnace Burden Development Section of the Department of Research and Technical Development have contributed to the design and have controlled the operation of the two plants, undertaken the experiments on the firing of the pellets, and have helped otherwise at all stages of the work. To these and other colleagues the author's thanks are due and, in particular, to Mr. P. E. Kersting who has also helped in the preparation of the manuscript.

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The Development of a Pelletizing Process for Fine Iron Ores

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SYNOPSIS

An account is given of laboratory and pilot-scale work on the pelletizing of Sierra Leone and Sydvaranger concentrates and on flash-roasted pyrites residues. Experience with a vertical-shaft kiln rated at ½ ton/hr. using external gas firing and incorporated solid fuel are related. Studies have been made of the structures of the pellets using microradiographic techniques. Economic aspects are considered in relation to the prospects of the process in the U.K.

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EXPANSION of the British iron and steel industry and increased consumption of imported ores have brought to the fore the problems of agglomerating fines before charging to the blast-furnace. Sinter-plant capacity, although continually increasing, has remained inadequate and the importation of fines and concentrates has made the situation worse. Concentrates present certain specific difficulties in sintering and in general reduce output, and the prospect of increasing proportions of these materials has caused some concern. For these reasons B.I.S.R.A. was invited to examine alternative methods of agglomeration with particular reference to the technique of drum pelletizing, being studied at the time in the U.S.A.

Laboratory work started four years ago and showed sufficient promise to justify the construction of a pilot plant to assess the technical problems involved and to give an approximate indication of the costs of the process in comparison with alternative methods, such as sintering. Drum pelletizing is not new, as shown by patent literature which includes such names as Andersson (1912), Stehli and Greenawalt (1920), and Brackelsberg (1931), but the current interest in the process seems to date from the work of the U.S. Bureau of Mines and the Mines Experiment Station at Minneapolis since about 1938. American interest in pelletizing has been mainly due to developments in the concentration of the taconites, a process which must result in a product in a much finer state of subdivision than normal iron-ore concentrates.

Although there has been a steady stream of notes in the technical press recently on commercial developments and prospects, the number of original technical papers has not been large. The first major publication from the Mines Experiment Station[1] was available when work was started in the U.K., and shortly afterwards an important Swedish paper[2] on laboratory work on the rolling process appeared.

MATERIALS STUDIED

When the investigation began emphasis was laid on processing hematite concentrates from Sierra Leone, which were reaching the U.K. in increasing quantities, and it was decided to base most of the more fundamental laboratory studies and, subsequently, the general development programme of the pilot plant on this raw material. Numerous factors would obviously require study and so there would be considerable advantages in confining attention to a single consistent material. Only much later, when the main lines of the technique had been worked out, were trials carried out on a variety of ore mixtures, magnetite concentrates, and pyrites residues.

LABORATORY STUDIES

Sierra Leone concentrates are derived from a semi-consolidated flaky hematite deposit and have the following typical analysis:

Fe, 66% (Fe_2O_3, 94%); SiO_2, 4%; Al_2O_3, 0·6%.

The size distribution is fairly narrow, with about 60% lying between 40 and 100 mesh:*

+ 60 mesh	− 60 + 100	− 100 + 200	− 200
35%	35%	25%	5%

Under the microscope the ore is seen to consist mainly of thin flat plates of hematite with minor quantities of conspicuous flakes of mica and irregular angular quartz grains. As received the ore is fine enough to impede the formation of a permeable bed in sintering, but it is much too coarse for balling and this is aggravated by the characteristic particle shape.

For the earlier stages of the laboratory work, therefore, small batches of concentrates had to be ground in laboratory mills until larger quantities in various size ranges became available from larger-scale grinding-test operations.

The laboratory drum system consists of a steel cylinder 19 in. long × 15 in. dia., closed at one end and provided with a 7-in. dia. hole at the other. The drum rests on a pair of 4-in. wooden rollers which drive it at 5–20 r.p.m. through a variable-speed gear

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* All screen sizes referred to are B.S.S.

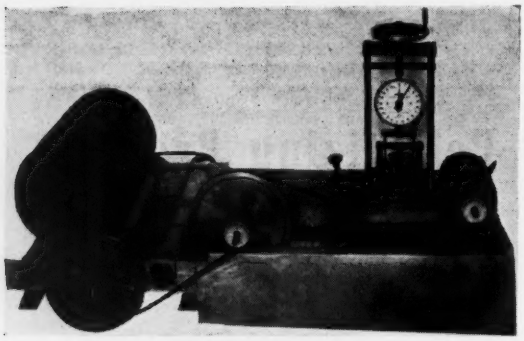


Fig. 1—Laboratory drum equipment

(Fig. 1). With the drum rotating, the ore is introduced with a scoop, and water or a binding-agent solution is injected from a wash bottle in a narrow stream. Dry or partially moistened ore can be used and it is easy to distribute ore and water over the tumbling mass in the drum as required.

A minor disadvantage of a drum of this size is that the normal charge weighs several pounds, so that excessive quantities of raw material are used up if, for example, an extensive series of tests is to be made with different binding agents. For such occasions a drum 6 in. long × 18 in. dia. was used, mounted directly on a variable-speed shaft. As a further alternative, the shaft could be inclined and arranged to support a 9-in. dia. metal bowl or even a 1000-c.c. flask from which the bottom had been removed, when even a single pellet could be formed under conditions approximating to the larger scale. With a little experience, moreover, it is possible to get an idea of the appropriate conditions for pelletizing by rolling a lump of the moistened powder in the hollow of the hand and adding dry powder until the superficial moisture disappears.

Initially, laboratory experiments were carried out with Sierra Leone concentrates ground to various size gradings and balled with water alone. It was soon found that there was a critical limit at about 50% in the proportion passing 200 mesh, below which pelletizing became difficult and slow. This figure (50% of − 200 mesh) usually corresponded to 80% of − 100 mesh and grinding to this specification by a variety of mills seemed to give essentially similar distributions. The optimum moisture content for pelletizing this ore was established as 8–10%.

Pellets were fired in batches of up to six in a Nernst–Tammann carbon-tube resistor furnace which would readily attain 1300° C. in 20 min. The furnace was lined with a 28-mm. internal dia. refractory tube into which the pellet or pellets were introduced before switching on. Oxidizing conditions were maintained throughout. It soon became evident that, with Sierra Leone concentrate pellets, the onset of consolidation in the firing was sudden. Pellets heated to 1270° C. were no stronger than those dried out at 100° C., a 1-in. pellet failing under compression at about 300 g. Heating to 1290° C. and switching off as soon as the pellet surface reached this temperature gave a product capable of supporting ½ ton and surviving repeated 50-ft. drops. The resulting pellet was fully oxidized and the mechanism of consolidation seems to be simple thermal recrystallization accompanied by surface diffusion (*see* p. 53).

The theory of the formation of pellets in a rotating drum has been discussed by Firth[1] and by Tigerschiöld and Ilmoni.[2] Firth suggests that compaction is a mechanical process brought about by extreme local pressure at minute points of contact between the drum and a small number of particles in the surface of the ball. Tigerschiöld has pointed out weakness in this explanation, which would demand a very smooth and inelastic drum surface. Moreover, such an effect could only influence the surface of the ball to a depth of a few particle diameters and, although a slow and continuous build-up of the ball is most effective in giving high strength, continued rolling of a soft aggregate can greatly increase the strength.

The present authors' experience leads them to support Tigerschiöld's theory of surface-tension forces directed towards the centre of the ball. On this view the main function of continued rolling is to confer mobility on the ore particles by a series of impacts directed towards the centre from all directions. Similar compaction can also be brought about by the surface-tension forces if a moistened mass of ore is subjected to vibration.

The fundamental requirement of the rolling process is to give the pellets the ability to survive the shocks of transfer to the kiln and the compressive stresses and abrasion while drying out and reaching the zone of maximum temperature. Various physical tests have been used at different stages in the work, mainly involving crushing and dropping, but when operations began on the pilot plant, reliance was placed chiefly on a compression test using the B.C.I.R.A. foundry-sand tester (*see* Fig. 1), coupled with visual estimates of the effect of dropping. A standard drop test is of doubtful value for two reasons. When the balls are dropped on to a hard surface such as steel or concrete a drop of 2–3 ft. usually brings about destruction immediately, whereas a very small drop of 2–3 in. is survived repeatedly. Over the intervening range, nearly all balls fail at the second drop. The other point is the profound influence of the surface on to which the pellet drops. Drops of 3 ft. on beds of soft pellets, rubber belts, or even wood cause little deterioration in a stream of green pellets.

By careful design in the handling system for green pellets the inherent shocks can be minimized, but once inside the kiln the dried (but as yet unconsolidated) pellets are subjected to mild pressure and a degree of abrasion depending on the smoothness of stock descent. With iron oxides of high purity and no binder, this seems to be the most critical stage in the operation, as pellets then have the lowest resistance to compression and shock. The function of a binder must be, in fact, to increase the dry pellet strength rather than the fired strength.

These observations brought to the fore the problem of the low strength prevailing in the dry pellet from 100° to 1270° C., and work was therefore undertaken to assess the effect of binding agents. A variety of organic substances, including starch, dextrin, sulphite lye, and alginate products, was assessed. In some

cases (*e.g.*, using up to 3% sulphite lye) an improvement in green strength, particularly resistance to impact, was observed, but none of these materials was found to improve matters in the most critical stage of the firing, because the organic substances were burnt out long before recrystallization bonding began.

Inorganic salts proved to be more effective, and an investigation which started as a study of the possibilities of increasing the surface-tension effect revealed a number of interesting features. The following typical determinations of crushing strength are indicative:

Additive	Solution Strength, %	Crushing Strength (1-in. Pellets) Green g.	Dried Rapidly g.	Fired at 1300° C. lb.
None	—	1000	300	1500
NaOH	2	1150	5400	2500
Na_2CO_3	3	1200	4600	3000
K_2CO_3	3	750	1500	—
$CaCl_2$	3	800	2000	2000
$MgCl_2$	3	1300	4000	700
$MgSO_4$	3	750	2700	—

From extended series of such tests it was noted that most of the salts used tend to raise the surface tension of water. This effect is not likely to be of great importance, although the concentration of salt will increase to saturation during drying-out. In several cases salts with a marked effect on the dry strength can exist as different crystalline hydrates. It is possible that successive stages of dehydration may result in an interlocking structure of small crystals.

The most important effect occurred with alkaline materials, of which the most practical is sodium carbonate.[3] Some reaction can probably occur between the free alkali and the silica in the ore, even in the cold. The possibility of the formation of sodium ferrite has also been examined, and attempts to extract this from dried pellets and to precipitate the iron gave faint indications, which were not, however, entirely conclusive. Tests on pellets and on briquettes made from chemically pure Fe_2O_3 confirmed that the presence of free silica is not necessary to produce the effect. With all materials so far studied, sodium carbonate is of advantage if the particle size distribution is below specification. Tests with sea-water

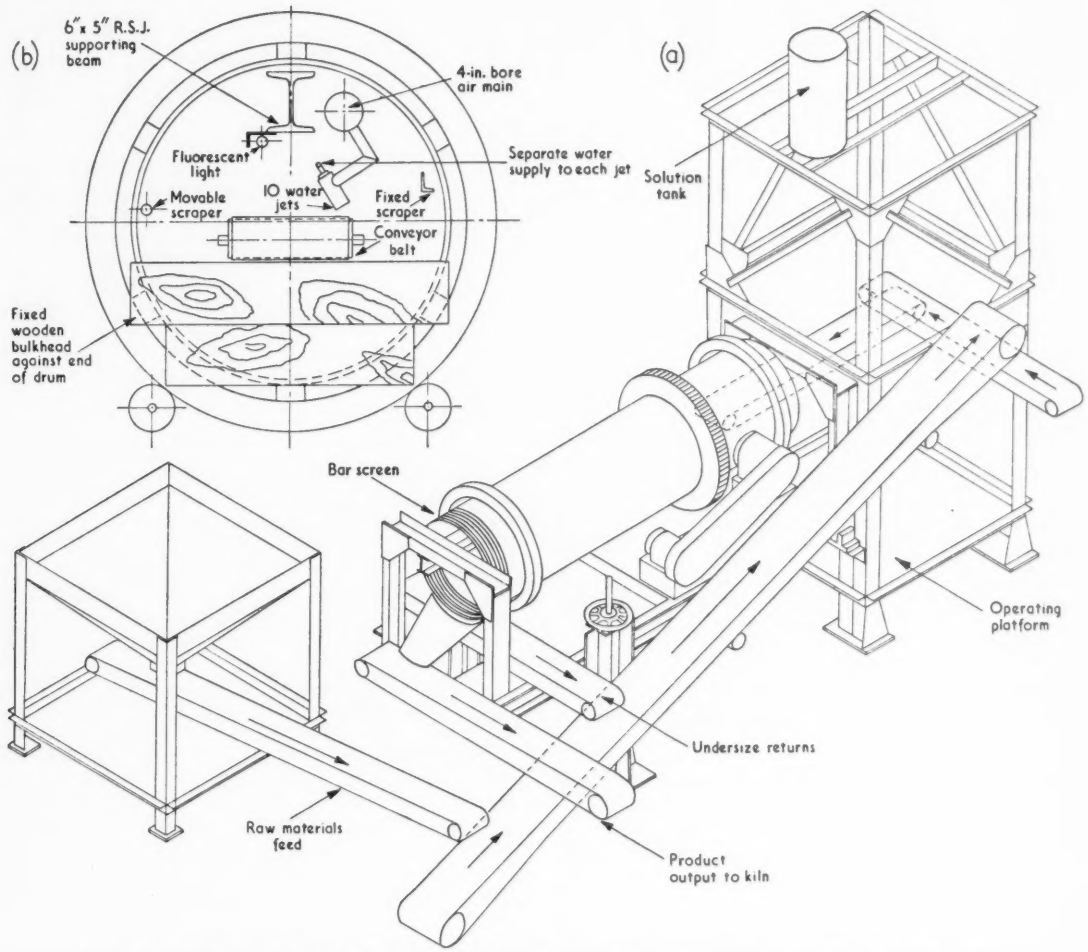


Fig. 2—(*a*) Isometric sketch of Redcar drum and conveyor system; (*b*) cross-section of drum

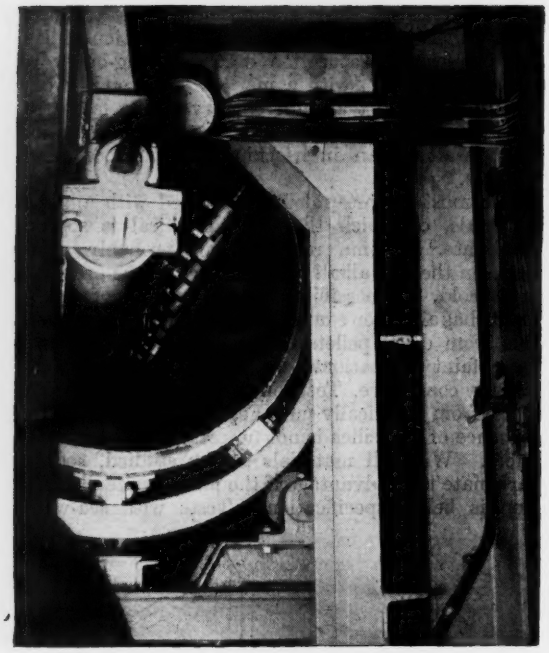


Fig. 3—Feed end of drum, showing spray units

showed a small improvement in dry strength. Although the effect is not very significant, it may contribute to the ease of pelletizing Sydvarangar concentrates which, it is understood, are washed with sea-water.

Although 50–60% of — 200 mesh must be attained in oxide materials devoid of natural coherence, relaxation is possible in the presence of clayey or otherwise sticky constituents. Tests were carried out at an early stage with the addition to concentrates of fines and slimes from British low-grade ores. To relax the size specification substantial (20%) additions were needed, and these caused slag bonding at a lower firing temperature. The resulting pellets were not so strong nor so reducible as those bonded by oxide recrystallization and, owing to the variable composition of home ores, optimum firing conditions were difficult to maintain.

Another series was worked out using mixtures of ground concentrates with imported ore fines. Wet-ground concentrates cannot be dewatered enough to pelletize directly (*see* Appendix I), and the aim was to add relatively dry fines in the hope of obtaining a mixture which could be pelletized and in which the moisture content was no longer excessive. Various fine ores are available with a moisture content as received of 4–5%, and 50 : 50 mixtures of these with ground Sierra Leone concentrates require 10–13% of moisture for pelletizing.

As an example, consider mixtures of ground concentrates with Sierra Leone fines (Fe, 60·25%; SiO_2, 6·5%; Al_2O_3, 3·4%). These contain very little material larger than $\frac{1}{8}$ in., and this fraction was first removed. Additions of fines were made in the range 0–50% and showed significant improvements in dry and wet strength. Maximum wet strength was recorded with 15% of fines, but dry strength increased up to 50% of fines when 1-in. dia. pellets withstood a crushing load of 20 kg. As the fines contain very little below 200 mesh, the — 200 mesh fraction in the mixed pellets was only about 30–40%. Essentially similar results were obtained with ore fines from North Africa, Spain, Liberia, and Sweden. The presence of $+\frac{1}{8}$ in. material was disadvantageous, as these particles failed to ball up completely and, for the most part, appeared in the drum output. With repeated circulation this fraction would eventually accumulate and choke the system. Although satisfactory pellets were produced in the experimental drum, there were occasional indications of difficulties which might arise on a larger scale. Thorough mixing of the constituents was essential and it was noted that on occasions irregular coating of the walls occurred. Pilot-scale tests using 75% of ground concentrates and 25% of fines were successful, but in comparison with 100% of concentrates the operation was less stable and rather more difficult to control.

DEVELOPMENT OF PILOT PLANT

In view of the success which was achieved in the laboratory, operations were transferred to a larger scale. Before proceeding to the design of a shaft kiln, it was thought desirable to consider the heat-transfer problem of heating pellets in counter current to hot products of combustion, especially as the initial material to be used was hematite, with which there appeared to be little experience elsewhere. The Math ematical Section of B.I.S.R.A. was therefore invited to examine the problem. The following conditions were proposed:

Final pellet temp.	1350° C.
Inlet gas temp.	1450° C.
Exit gas temp.	200° C.
Pellet dia.	$\frac{1}{2}$ in.
Cross-sectional area of shaft	2·2 sq. ft. (20 in. dia.)
Throughput	1000 lb./hr.
Fuel: Blast-furnace gas with 25% excess air at 800° C.	

These conditions did not in all cases correspond with those actually used, but were sufficiently indicative. The resultant analysis, given in Appendix II, shows that conduction in the pellets imposes a negligible restriction in comparison with the gas/solid heat transfer. The requisite bed height deduced was only 2 ft. with 800 lb./hr. hot gas flow. This result is remarkable, but evidently in accord with U.S. experience. It was used in designing the pilot kiln with success.

For the pilot installation, an existing building* 46 ft. × 36 ft. × 22 ft. high was used. It was decided to aim for a throughput rate at all stages of $\frac{1}{2}$ ton/hr. All sections of the equipment were to be as adaptable as possible to permit experimentation over a wide range of conditions.

The drum-balling system was originally devised to allow for alternate spraying and dusting with dry ore as the pellets moved along the drum. This was intended to give maximum flexibility in studying the

* Made available by Dorman, Long and Co., Ltd., at their Redcar works.

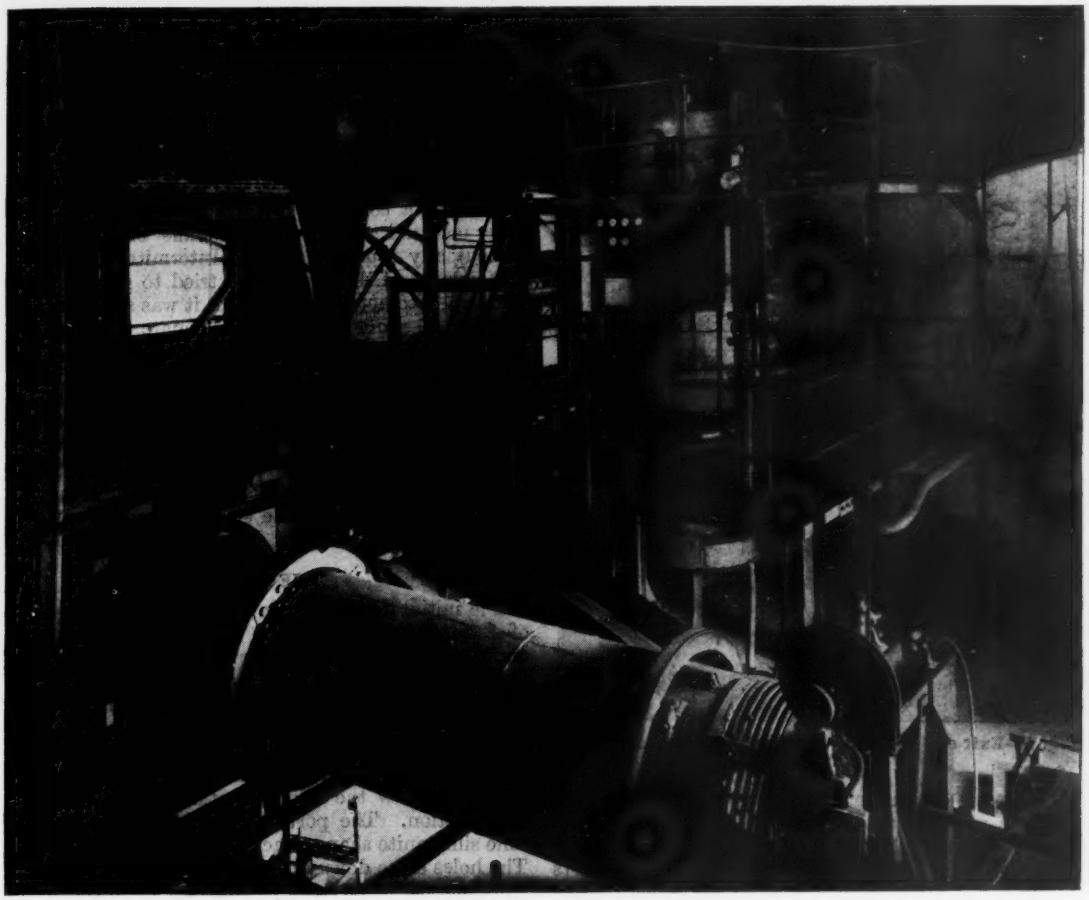


Fig. 4—General view of pilot plant

build-up of pellets. Dry grinding of the concentrates was used largely for reasons associated with delivery available on various alternative grinding plants, and it seemed logical to aim for more than one stage of build-up of the pellets in a single passage through the drum, thus reducing the circulating load. Although this principle was later abandoned, it led to a rather large length/diameter ratio. Subsequent experience has shown that the additional length is an advantage, as it prolongs the rolling time beyond that required for building up additional layers on the pellets and this improves the structure of the outer layers.

The drum (2 ft. 9 in. internal dia. × 11 ft. 3 in. long) is mounted on a cradle (Fig. 2*a*) supported on hinges at one end and screw jacks at the other, so that the angle of inclination of the drum can be varied from horizontal to 2½° (½ in./ft.). A 5-h.p. variable-speed drive unit is also carried by the cradle, and allows the rate of rotation of the drum to be varied from 4 to 14 r.p.m., corresponding to 35–120 ft./min. peripheral speed. A speed of 10 r.p.m. (= 86 ft./min.) has been found most suitable. All internal fittings of the drum are supported on a 6 in. × 5 in. I beam located a few inches from the top (Fig. 2*b*). This beam carries the water-spray system, consisting of ten atomizing units at 8-in. intervals, starting from the entry end. The atomizers are fed from an air main connected to a fan, giving 50 cu. ft./min. at 4 in. W.G. The supply of water or solution to the atomizers is individually piped and controlled. Three sprays are normally sufficient and the remainder are now redundant. Each spray is adjustable for axial direction, intensity of spray cone, and droplet size, The internal beam also supports two 80-W. tubular fluorescent lamps. A fixed scraper is provided for the whole length of the drum.

Figure 3 shows the arrangement of the sprays (the worm conveyor shown is no longer used). Figure 4 is a general view of the plant and Fig. 5 is a view of the exit end, showing the screening arrangements.

Essential features of the balling system are adequate screening facilities and provision for feeding the undersize back into the drum. Much development work was necessary to arrive at a satisfactory solution, because most of the raw materials, with a moisture content in the neighbourhood of that required for pelletizing, will not run down chutes, however steep. The most effective screening arrangement is a spiral-bar screen forming an extension to the drum itself. Undersize material falling through the screen is taken

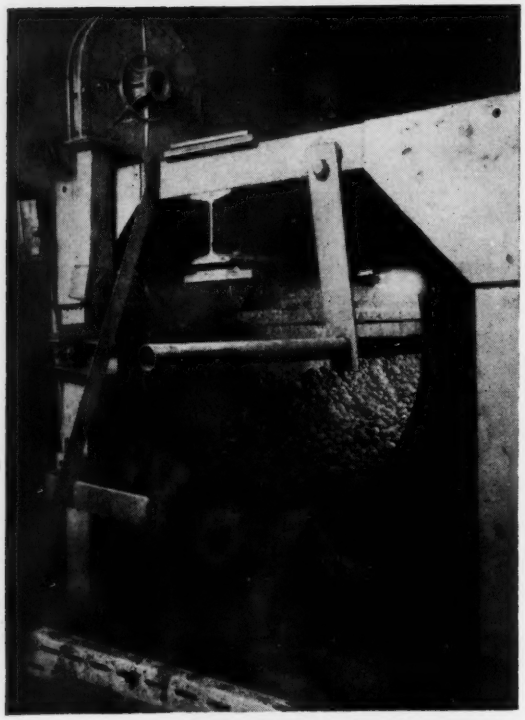


Fig. 5—Exit end of drum, showing screening arrangements

by a system of four conveyors with three right-angle transfer points into the head of the drum. The finished oversize pellets fall on to an output conveyor. Although it is possible to produce very large pellets (*e.g.*, 4 in. dia.) the system may be controlled to give a narrow size range in the output without having to screen out oversize for breakage and return. With the multiple conveyor system for returns, new feed can be added at any convenient point in the circuit.

From time to time different spacings have been used in the spiral-bar screen. With Sierra Leone concentrates a $\frac{3}{4}$-in. spacing was first used, and this gave a range of product size of $\frac{3}{4}$–$1\frac{1}{2}$ in., with an average of about $1\frac{1}{4}$ in. A reduction in spacing to $\frac{1}{4}$ in. became necessary for the concentrate–fines mixtures, and subsequent work has been carried out with $\frac{1}{2}$-in. spacing. This size seems best in most cases and gives an average product size of 1 in., with a small proportion of pellets up to $1\frac{1}{2}$ in.

The proportion of returns varies with operating conditions and may be as little as 30% with a high rate of output using a feed with 60% or more below 200 mesh.

During the experiments it became evident that the amount of material rolling in the drum had a marked effect on performance. With a small load, most of the fresh material was not picked up by the circulating pellet nuclei, but rather formed into small pellets. This gave rise to very large circulating loads and low output. To increase the mass of tumbling material the slope of the drum was reduced to zero and the feed was increased. Spillage of material from the input end was prevented by introducing a wooden bulkhead pressed against the rim of the drum at this end. These changes effected a marked improvement and, together with increasing familiarity with the process, led to an increase in output from an initial rate of $\frac{1}{2}$ ton/hr. to over 3 tons/hr., partly depending on the material being processed.

In the early stages of operation there was a tendency to develop a condition with a mass of pellets and loose material sliding on the walls of the drums without any tumbling effect. Intermittent scrapers and rapping of the drum were tried to give a rough surface to promote tumbling, but it was subsequently found that, provided that an adequate drum charge was maintained, use of a fixed scraper alone was sufficient. The degree of roughness of the surface is undoubtedly of great importance, and the fixed scraper gave a coating just rough enough to maintain the tumbling action. Vibration of the scraper, because of its length, is probably instrumental in attaining this, and the effect of water addition near the feed end enhances the slight irregularity of the surface at this point.

THE KILN

The initial design of kiln involved a parallel shaft of 2 ft. internal dia. with ports leading from an annular combustion chamber and entering the shaft between levels 4 ft. 8 in. and 5 ft. 9 in. from the top. Although the main body of the structure has not been disturbed, various modifications have since been made to the design of the ports, and the section above this level has been tapered and shortened. Figures 6*a* and *b* show the kiln in section before and after modification. The port-wall section is made up of nine sillimanite shapes, each with ten 1-in. dia. holes. The holes slope downwards and are further protected from ingress of the stock by overhanging lips (Fig. 7). The remainder of the hot-face brickwork and the combustion-chamber lining is of 50% alumina brick and is backed by high-temperature insulating brick. The whole structure is steel-encased, the brickwork design being based on a calculated temperature of rather less than 100° C. at the shell. A large number of thermocouples (originally 20) were provided, together with nine refractory access tubes for use with total-radiation pyrometers and pressure gauges. All these introduced weaknesses into the brickwork and the consequent circulation of hot gases gave rise to local overheating of the shell, which had to be controlled by water sprays. This has had an adverse effect on thermal efficiency, which should be borne in mind when assessing fuel requirements.

Coke-oven gas is used for pellets containing no solid fuel, and this is burnt in a single Schieldrop turbulent burner from which it enters the annular combustion chamber tangentially. Gas pressure is controlled at 8 in. W.G. and combustion air is supplied by a fan rated at 300 cu. ft./min. at 10 in. W.G. The combustion air supply is controlled by a Kent proportional + reset (integral) Multelec Mark XXI instrument to maintain a constant temperature in the combustion chamber. Two 6-point Kent recorders can be connected as required to selected thermocouples in the brickwork. Static pressure indicators are con-

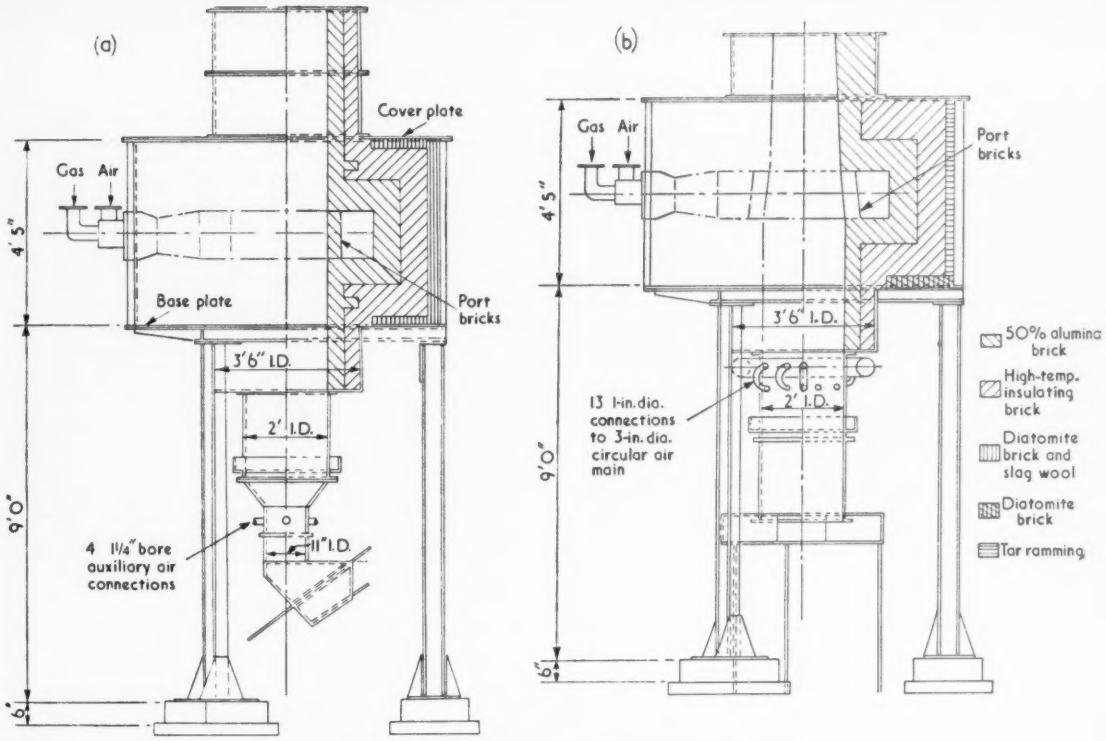


Fig. 6—Shaft kiln: (*a*) First design; (*b*) modified design

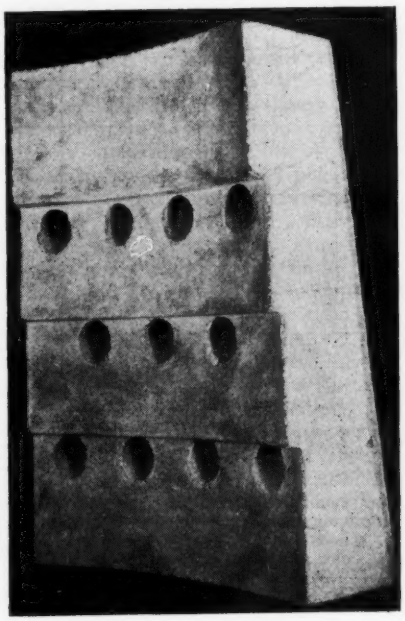


Fig. 7—Port block

nected to points in the stack and combustion chamber.

The small scale of the plant imposed restrictions on the design of the discharge mechanism. The basic requirements are that discharge shall be substantially continuous, that masses of pellets fused together by accidental circumstance can be broken up or otherwise extracted, and that air and gas leakage shall be controlled. The present arrangement does not provide for continuous discharge, but is reasonably satisfactory for the scale of operations. It consists of a circular table supported 6 in. below the bottom of the kiln shaft, the whole being cased in with four access traps from which the pellets can be removed by shovel according to timetable (Figs. 4 and 6*b*).

Although it had been hoped to arrange belt feed to the kiln, site restrictions made it necessary to use batch handling, the green pellets being collected in buckets from the output belt and hoisted to the top platform. The rather rough handling involved has necessitated re-screening on a ½-in. screen before charging to remove fragments which might otherwise decrease the permeability in the shaft.

OPERATING EXPERIENCE

Numerous experiments have been carried out on the pilot plant under gas-fired and solid-fuel-fired conditions using a variety of raw materials. The limitations on materials-handling facilities and staffing have prevented the performance of trials of long duration, and in practice 12 hr. has proved to be an upper limit to the operations. Instrument records

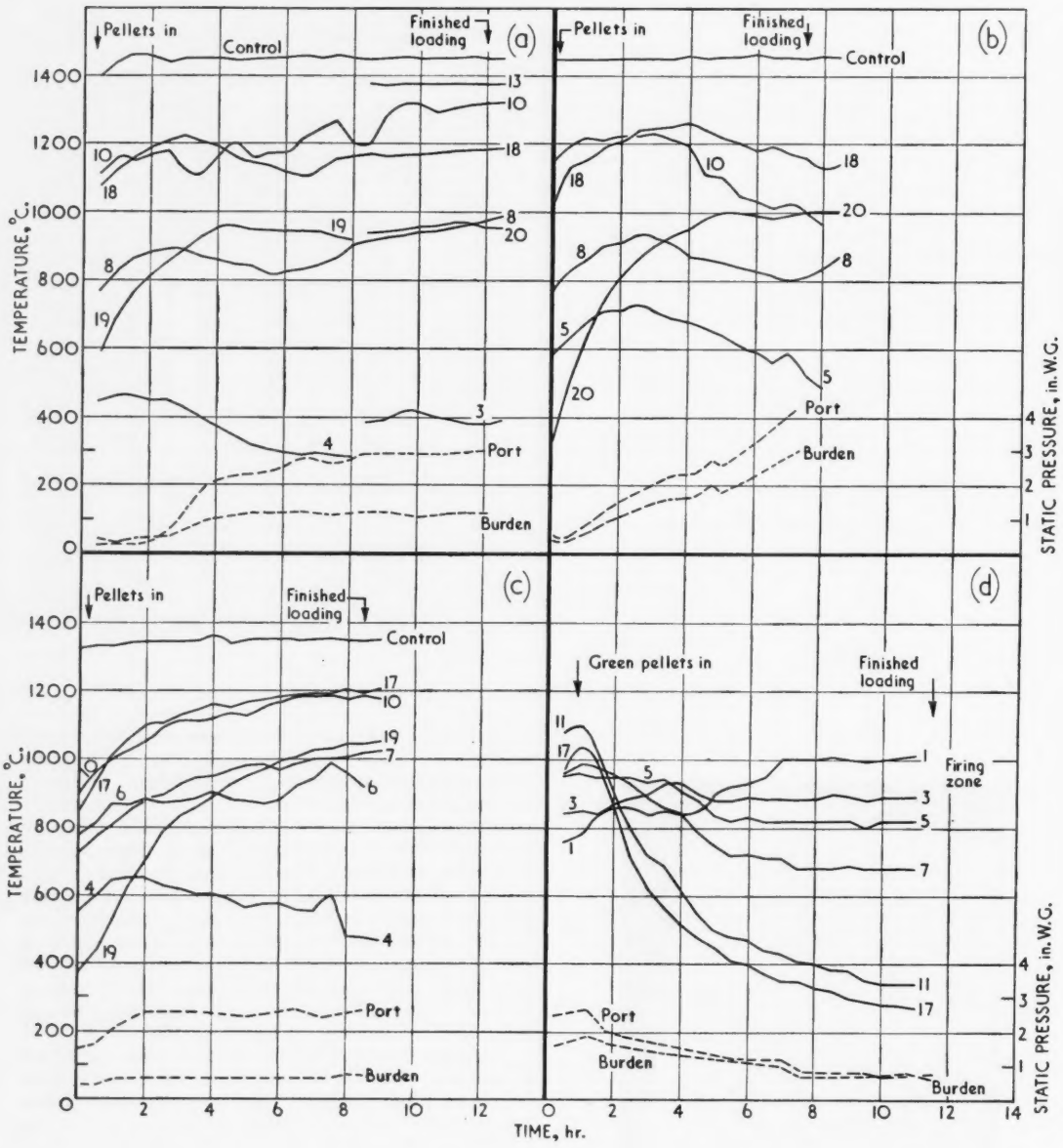


Fig. 8—Chart records of trial runs: (*a*) and (*b*) Sierra Leone concentrates, gas-fired; (*c*) flash-roasted pyrites, gas-fired; (*d*) Sydvaranger concentrates with 2% coke addition

suggest that steady conditions are usually established in about 6–8 hr.

The following selection has been made to show the type of difficulties experienced and typical data for the conditions of the pilot plant.

Gas-Firing Trials

Gas-firing trials with Sierra Leone concentrates were carried out in the early stages of the development and were therefore subject to two inconveniences which no longer apply. The drum and raw-materials-handling system was still under active development at the time and delays and stoppages were frequent. It was therefore necessary to maintain a large stock of green pellets on the ground, and the recovery and re-screening of these resulted in the charging of an undesirable proportion of broken pellets. The other weakness at this time was that the kiln shaft had no batter, a fact which tended to encourage sticking, and this was aggravated by the awkward discharge arrangements of the time (Fig. 6*a*).

For starting up, the kiln was filled with fired pellets and gas was put on to raise the combustion-chamber temperature slowly. When this reached 1100° C., slow withdrawal of the pellets was started and continued, and the combustion-chamber temperature was raised to the required value of 1450° C. Fired pellets were introduced to keep the stockline constant. Shortly before the required firing temperature was reached, screened green pellets (¾–1½ in.) were loaded and the withdrawal rate was adjusted to 1000 lb./hr. A small volume of auxiliary air (50 cu. ft./min.) was maintained to cool the pellets before discharge; this air escaped through the discharge and was not available for recuperation.

The results from two trials, one 12 hr., the other 8 hr., are shown in Figs. 8*a* and *b*. The volume of air admitted to the burner varied between 200 and 225 cu. ft./min. with a coke-oven-gas consumption of 27–28 cu. ft./min. (net calorific value 463 B.Th.U./cu. ft. saturated at 60° F.). This gave a combustion-chamber temperature of 1450° C. and, as the excess air was about 70%, fully oxidizing conditions were maintained. The top gas temperature varied between 100° and 175° C., and the fired pellets were discharged at about 600° C. The graphs represent the combustion-chamber temperature and inwall temperatures at various levels in the kiln and the back pressure at the ports and at a position in the kiln inwall above the ports. A period of 3–4 hr. was required before conditions became uniform. Thereafter, the inwall temperature above the ports showed the greatest fluctuations, caused by the intermittent burden descent obtained with manual unloading.

In Fig. 8*a* very steady conditions are shown after 9 hr. operation, and the run was only brought to an end by exhaustion of available raw materials supplies. Figure 8*b* shows a markedly different state of affairs, with a continued increase in resistance to gas flow, as indicated by the back-pressure figures, and a decline in operating temperatures throughout the heating zone. Under these conditions considerable losses of combustion gases occurred through thermocouples and other access points in the brickwork, and throughput rates had to be limited to ensure consolidation of the pellets. At the end of this test it was found that the ports had become partially blocked.

Scaffold formation at a point just above the ports occurred on several occasions. This formation usually consisted of an agglomeration of fine material and pellets fritted together, the layer immediately adjacent to the inwall being almost wholly fines. The scaffold was easily removed on cooling the kiln, and the brickwork showed little sign of attack. Factors contributing to these formations were the intermittent discharge, the introduction of fines on loading, the abrasion of pellets on the kiln wall, and the parallel lines of the kiln. There was also a tendency for the green pellets to bridge on loading. This bridge collapsed when the burden underneath had been lowered, and some breakdown of the semi-dry pellets resulted. This trouble became more serious when self-firing with coke addition, but was later overcome by tapering the kiln from the ports to the stockline.

More recently, another series of trials was undertaken using gas-firing, and by this time the kiln had been modified by the incorporation of special port bricks. The brickwork of the upper shaft had also been replaced, so that there was a continuous taper from 2 ft. dia. at the base of the ports to 1 ft. 6 in. dia. at a distance 1 ft. 6 in. below the stockline; the first 1 ft. 6 in. thus remained parallel. After causing some trouble with hanging at the stockline, this parallel was removed to give a total distance from the top of ports to the stockline of 3 ft. 6 in. Flow of materials down the shaft was much improved, but a continuation of the expanding portion of the shaft to about 2 ft. below the ports remains most desirable. The other major improvement was the enclosed-table type of discharge shown in Fig. 6*b*.

The later trials were carried out using residues from the flash-roasting of pyrites. This material, which approximates to pure Fe_2O_3, arises in an extremely fine state of sub-division, about 65% passing through 200 mesh. Its use in sinter plants is therefore difficult, and the disposal of the large and increasing production by pelletizing is worthy of study. Flash-roasted residues may be expected to carry about 1% of sulphur, ¾% of copper, and ½% of zinc, varying according to the source of the pyrites, and significant differences in behaviour on firing have been noted. Residues from two sources have been examined and laboratory studies have suggested appropriate firing temperatures of 1100° C. for Kalavassos residues and 1250° C. for C.M.C. Solea.

For the pilot-scale tests the kiln was operated as for Sierra Leone concentrates. Only one short trial was completed using Kalavassos cinders, and well-fired pellets were produced with the combustion-chamber temperature controlled at 1150–1175° C. A number of trials were made with C.M.C. Solea with the combustion-chamber temperature at 1300–1325° C. The volumes of air and gas were rather low in this trial (215 and 22·5 cu. ft./min., respectively) and a throughput of only 4½ cwt./hr. could be maintained. Figure 8*c* shows the corresponding temperature and pressure records.

In assessing the results of these trials with low throughput, it should be borne in mind that the cross-

kiln had been reduced, the stockline me case) by as much as 44%. In moisture content of the pellets was

Solid Fuel Firing

Solid-fuel firing has a number of attractive features. In this practice a suitable proportion of finely divided coke or coal is mixed with the raw-ore-fines feed and thus incorporated in the green pellets. The green pellets are then burned in an updraught of air in a shaft. As the air stream first traverses a bed of fired pellets, recuperation is excellent and quite small quantities of fuel are sufficient to provide a temperature high enough for consolidation and hot gases for preheating and drying. Penetration of air to the cores of the densely packed pellets and escape of the products of combustion is remarkably easy, and a concentrated hot zone less than 1 ft. high results. In the case of hematite pellets some reduction to magnetite must occur in the presence of carbon in the high-temperature zone but, under favourable conditions, re-oxidation is substantially complete. The process is not without disadvantages, however. The proportion of fuel is naturally rather critical, and there is a long time lag between the point of addition before the pelletizing drum and the combustion zone in the kiln. Good mixing is essential, as each pellet must contain a close approximation to the optimum amount of fuel (about 1 g. for a 1-in. pellet). To attain good recuperation, the air must be introduced low in the shaft; with the pilot-kiln arrangements some of the air must enter at the ports only 4 ft. below the stockline. The difficulties of adequately sealing the discharge mechanism were also a drawback, as an excessive proportion of the air was lost there. In a commercial plant it would be essential to provide an adequate seal, or alternatively, to include the entire discharge mechanism within a forced-draught system. The level of the combustion zone is determined by a balance between the rate of air supply and the rate of stock descent, and commercial operation will demand some ingenuity in controlling one or both of these rates in relation to some index of combustion-zone location. On the pilot plant, where appropriate thermocouples gave a useful indication, the zone was maintained at a high level. On occasion it was even allowed to rise to a point at which it was visible from the stockline from time to time. Surprisingly, little degradation occurred when charging green pellets on a hot stockline.

Early work was again concerned with Sierra Leone concentrates, coke being added during grinding to ensure thorough mixing. The carbon content of the mixture was checked by analysis.

When starting up, a full charge of previously fired pellets was heated until the temperature of the pellets at the stockline reached about 800° C. Pellets were then rapidly discharged to lower the stockline 18 in. and a layer of fired pellets was loaded, together with a little coke breeze. Green pellets were then charged until the stockline was recovered. The pellets were then drawn through at a predetermined rate, the firing zone being maintained about 2 ft. below the stockline. The coke-oven gas was cut off 15 min. after the addition of the green pellets, and thereafter air was blown through the ports (225 cu. ft./min.) and from the auxiliary main (75 cu. ft./min.).

Preliminary trials showed that 3% of carbon was the correct proportion for self-firing. Good pellets were produced, but there was a slight tendency to form agglomerates of pellets and fines, which were troublesome to discharge. The introduction of a clinker breaker is beneficial, as the agglomerations are easily disintegrated. A try rod introduced into the burden showed the flame front to be 2 ft. below the stockline and the high-temperature zone to be 10 in. deep. The flame-front speed was about ½ in./min. and the throughput just over 925 lb./hr.

At this time the question of sintering wet ball-milled concentrates arose and the effect of addition of proportions of Sierra Leone fines to the mix was examined. Opportunity was taken to modify the kiln by installing a new auxiliary air main and the table discharge; 3% carbon addition was again used. The ratio of air blown at the ports and auxiliary mains was varied, but the total volume was maintained between 315 and 385 cu. ft./min. The pellets produced were inferior to those obtained with concentrates alone.

Shortly after the kiln modifications, Sydvaranger concentrates became available for use, and as these did not require grinding it was decided to carry out trials and incorporate ground coke for self-firing. To minimize escape of air at the discharge table slight modifications were made to the starting-up procedure. A blank of fines 1 ft. deep was loaded below the ports to seal the bottom section of the kiln and the kiln was heated as before. In this case the firing zone was raised to 6 in. from the stockline.

Additions of 3% of coke were first tried and these pellets were fired successfully, but a central 9-in. dia. column of fused pellets was found to extend from the discharge table to the firing zone. It had also been noted that the uppermost layers of pellets in the drying zone showed a slight tendency to stick together. The proportion of fuel was therefore reduced to 2% and experiments were carried out with a central refractory plug suspended in the stockline region. Pellets were charged in the annulus thus formed; as they descended to reach the unobstructed section of the shaft the rolling action to the central axis was effective in separating pellets tending to cluster. Such an arrangement was useful in overcoming the disadvantages of the parallel section at the stockline and satisfactory runs were obtained. Figure 8*d* shows the inwall temperature and static pressures. The temperatures after initial variations reached stable values after 6 hr. The static pressures fell as the blank was removed and thereafter remained uniformly low. The volume of air blown was 220–235 cu. ft./min. through the ports and 85 cu. ft./min. through the auxiliary main. The flame-front speed altered from 1 to ½ in./min. as the fines blank was removed, clearly showing the loss of combustion air through the open discharge.

Balling up green pellets and firing them to give a good-quality finished product was very easy with these concentrates. This was no doubt due to the very fine particle size (86% through 200 mesh) and also to the exothermic oxidation of the magnetite.

Trials were similarly conducted with flash-roasted pyrites cinders. It might have been expected that the heat of combustion of the residual sulphur in the material would have allowed a reduction in the proportion of coke required, but 3% was found to be necessary. With Kalavassos residues the strength of the fired pellets was low, and large variable proportions of dust were obtained. Satisfactory results had not been achieved when the source of supply was changed. More satisfactory results were obtained in work with C.M.C. Solea residues.

It was established that 3% coke addition was correct for this material. A thermocouple inserted 2 ft. into the top of the burden was used to control the rate of discharge. A maximum temperature of 1290° C. was recorded on this couple. The combustion air blown was 100 cu. ft./min. through the ports and 150–185 cu. ft./min. through the auxiliary main. The pellets produced were fully fired, but were not quite as strong as those from Sydvaranger ores; they appeared to be duplex, consisting of an outer sintered skin separated from a fused inner core.

Flash-roasted cinders present an interesting possibility of using finely ground unfired pyrites as a fuel with recovery of sulphur oxides from the waste gases from the kiln. Batches of green pellets, made with 9 and 18% of raw pyrites, respectively (sulphur content about 50%), were charged at the end of a normal run. In both cases extensive disruption of the pellets occurred as the combustion zone was reached; this work has not been pursued further.

MICROSTUDIES OF PELLET STRUCTURE AND TEXTURE

The laboratory and pilot-plant pelletizing trials were supplemented by extensive investigations of the microstructure of pellets. It was thereby hoped to obtain some information on fundamental principles of pelletizing and to exert closer control over the larger-scale trials.

Such studies require a technique permitting relatively rapid examination of large numbers of pellets without undue expenditure of time or effort in preparing the samples. The use of polished surfaces was rejected as difficult and time-consuming, and also because too many grains were pulled out of the sample surface, giving a false impression of porosity. Ordinary thin sections (about 25 μ thick) were liable to suffer from similar shortcomings, and it was decided to use a combination of thick section (50–60 μ) and microradiographs.

Microradiography had been used successfully in the study of iron ores[4] and sinters[5] and was found to give speed and simplicity to the present work. Pellets were sliced in half with a high-speed diamond saw. The cut surface of one half was impregnated with Canada balsam, the impregnation usually penetrating to a depth of about 100 μ. The surface was then ground flat and mounted on a thin cover glass, which in turn was cemented to a normal slide glass. The specimen was then sliced again on the diamond saw, leaving a section 60–70 μ thick on the glass carrier. The fresh surface was lightly ground, but no high polish was attempted. The thin cover glass carrying the specimen was detached from the slide glass and mounted in a suitable plate holder, the pellet slice surface being in contact with the emulsion of a photographic plate. Exposure to suitably selected X-radiation produced an absorption shadow photograph which could be studied under the microscope. The pellet slice was subsequently mounted on a slide glass for direct microscopic study of transparent minerals and for storage. Direct microscopic study provided little information apart from optical identification of crystalline slag phases formed during the firing process.

Part of a typical pellet radiograph is shown in Fig. 9*a*. The pellet consists of pure Sierra Leone hematite concentrates ground to 60% — 200 mesh B.S.S. The ore particles, white or grey in the radiograph owing to their absorption of the X-rays, range in length from about 0·15 mm. down to very fine sizes of 1 μ and less. A rough parallel alignment of the long axes of larger particles can be observed, the latter lying approximately perpendicular to the pellet radius, but the considerable enlargement somewhat obscures the concentrically layered pellet structure. Pores and voids in the pellet appear dark grey or black on the radiograph, and it is seen that the pellet has a very fine sponge-like texture. Many of the larger holes in this pellet were probably produced during cutting, but quite large cavities can be formed under certain conditions. Measurements on large numbers of pellets have established an average porosity of 25%, and Sierra Leone hematite pellets are superior in this respect to pellets made from magnetite concentrates. The high porosity was surprising at first sight because the flat flaky particle shape of the hematite was expected to result in very dense packing. However, close inspection of Fig. 9*a* shows many particles partly outlined by narrow black edges, *i.e.*, holes or gaps between the particles and their surroundings. These gaps are regarded as being due to shrinkage on recrystallization of the finer particles, which were packed against the larger flakes during the rolling process. The perfect and smooth crystal surfaces of the larger flakes offer few points of attachment during diffusion-recrystallization and large flakes tend to become attached to their surroundings, mainly along their broken edges. Shrinkage, which accompanies recrystallization of the finer powder, will therefore tend to pull the latter away from the less active flake surfaces. The gaps thus formed probably contribute much to the high porosity of the pellet.

Provided that the pellets are fired correctly, *i.e.*, under oxidizing conditions and between 1280° and 1350° C., the original particle shape and the layered structure of the pellet will be largely preserved and the porosity will be of the high order quoted. Reducing conditions during firing will result in the pellet structure shown in Fig. 9*b*. An accidental reduction in air supply occurred while this pellet was being heated in a gas-fired furnace. The hematite flakes have been partially reduced and have recrystallized to magnetite, which is seen to form an equigranular closely-packed aggregate of relatively low porosity.

Recrystallization to magnetite is always accompanied by compaction and considerable shrinkage in pellet volume. The pellets become brittle without

increase in crushing strength and the porosity drops considerably, depending on the extent of the reduction. Reduction proceeds from the surface towards the centre of the pellet and all stages between a thin outer skin of magnetite on unreduced hematite and a completely reduced pellet may occur. Reduction is often accompanied by localized partial fusion of the pellet followed by the growth of secondary dendrites of magnetite (Fig. 9*c*). Under conditions of more serious reduction, surface fritting and caking together of pellets occur, which may lead to serious scaffolding in the kilns. Reduction will also cause attack of the refractory lining and firing under highly oxidizing conditions is essential, not only for the quality of product but also for the continuity of the operation.

The pilot-plant trials included series of tests where external gas firing was replaced by coke incorporated in the pellets. This ' internal ' combustion invariably caused at least partial reduction of the pellets, and the increased proportions of slagging constituents due to the coke commonly resulted in fusion of the pellet cores. It was nevertheless possible to operate the kiln quite satisfactorily, provided that a good supply of excess air maintained overall oxidizing conditions. Under such circumstances pellets were found to have an outer layer of unreduced and relatively unaltered hematite. This is followed by a zone of increasing reduction characterized by a gradual increase in magnetite and corresponding disappearance of the original layered structure. Small globular slag pools full of minute secondary dendrites make their appearance and the texture and grain size of the magnetite coarsen noticeably. The centre of the pellet is occupied by very coarse crystals of magnetite embedded in crystalline slag or by a cavity lined with well-shaped crystals.

Figure 10*a* shows the margin of a coke-fired pellet. The pellet is more cavernous than a gas-fired one, owing to the holes left by the burnt-out coke and the gases evolved from the latter. This emphasizes the need for really good mixing and distribution of the coke, so as to minimize weakening of the pellet structure. The general flake shape of the hematite is still well preserved here, although a rounding of many of the flakes can be seen.

Figure 10*b* refers to a position further in from the margin of the pellet, but is still mainly in the outer zone. Large cavities clearly indicate bad coke distribution. A sudden change may be noted near the bottom of the radiograph, where the texture becomes dense as the margin of the inner zone is reached. The microscope shows here minute well-shaped crystals of magnetite, but reproduction has obliterated much of the finer detail. The dense texture is not accompanied by any evidence of sudden shrinkage, and may be due to compaction under gas pressure from burning coke near the core of the pellet.

Figure 10*c* is nearer the centre than Fig. 10*b*. The crystals of magnetite are coarser and are accompanied by slag pools with secondary dendrites of magnetite. A clear distinction should be made between the large crystals of magnetite, which are formed by recrystallization of hematite in the solid state, and the dendrites, which have crystallized from material taken into solution in the slag.

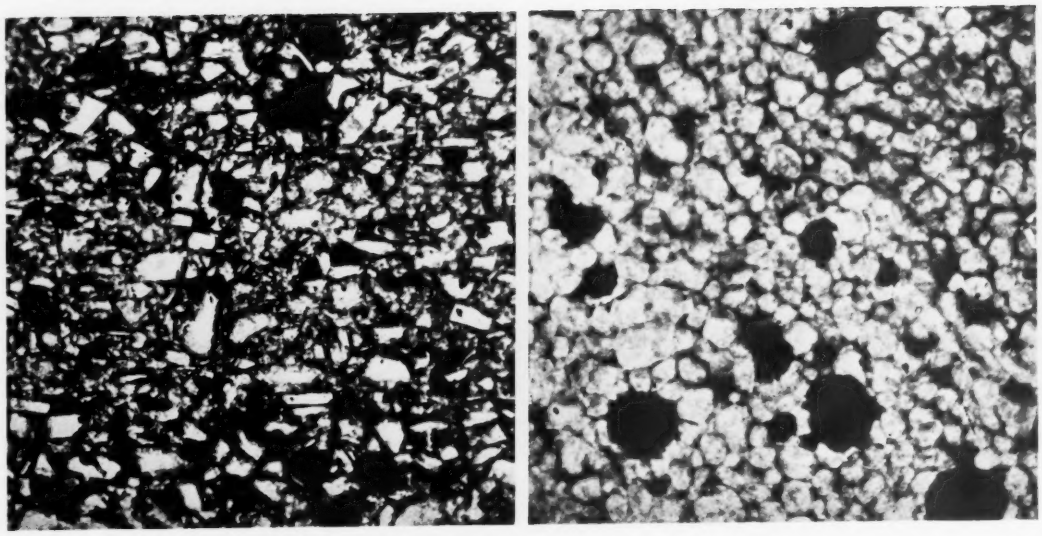
Figures 10*d* and *e* show the portion nearest the large central cavity of the same pellet. Magnetite crystals of up to 4 mm. dia. have been measured in this zone; the crystals are often well-shaped octahedra and are embedded in a crystalline iron-bearing slag which consists mainly of an iron-rich olivine near fayalite. All the large slag areas in Figs. 10*d* and *e* are occupied by tightly packed lath-shaped and fibrous crystals of this mineral. The radiograph does not show the outlines of individual slag crystals very clearly, but some indication of the fibrous structure can be seen. The light grey colour, intermediate between the white of the magnetite and the black of holes, indicates X-ray absorption due to the iron content of the slag mineral. Near the lower margin of Fig. 10*e*, the actual edge facing the central cavity can be seen. These cavities are common to coke-fired pellets and may be roughly rounded or irregular and flattened in shape. Projecting into the cavity are often found perfectly developed crystals of magnetite ranging in size up to 5 mm. side length. These crystals must have grown very rapidly, because each pellet passes through the kiln in 5 hr.; only 2 hr. of this time is spent at an elevated temperature, and not more than ½ hr. above 1200° C. Figure 11 shows half of one large cavity where many crystals display skeletal or hopper faces, generally regarded as an indication of rapid growth.

These man-made crystals are detrimental in pelletizing. Such pellets, slagged and containing zones of high density, show low porosity, low reducibility, and often relatively low physical strength compared with fully oxidized pellets of similar size fired by external combustion of gas or oil. Firing pellets by means of incorporated coke breeze or coal dust will probably only be attractive if an appreciable reduction in cost is thereby attained.

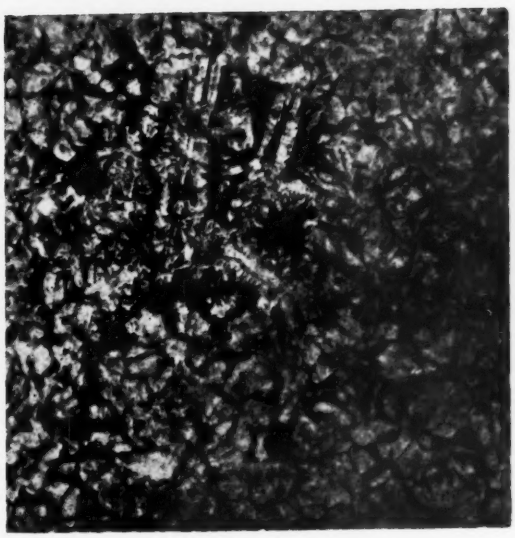
The remarkable improvements in unfired pellet strength obtained through the use of dilute solutions of certain inorganic salts might have been expected to be accompanied by some visible changes in pellet structure or texture. Large numbers of pellets were studied, but no changes could be detected that were definitely attributable to the use of one binder or another. In a selection of pellets made with plain water and various binders, it was impossible to note any specific peculiarities. In contrast, variations in pelletizing conditions (*e.g.*, proportion of very fine particles or speed of rolling) caused definite differences in pellet texture.

For example, Fig. 12*a* shows part of a radiograph of a pellet which was first built up from material containing neither very coarse nor very fine size fractions. The absence of fine material has caused the coarse loose texture seen in the lower part of the figure. Such pellets always have a low crushing strength, both unfired and fired, and are prone to abrasion losses.

Build-up of the pellet was continued with material rich in very fine size fractions. Although subsequent firing has obliterated the individual small particles, the much denser texture of the outer layer is clearly visible in the upper part of Fig. 12*a*. The build-up of this pellet was fairly rapid, the rate of adding material being kept high in relation to rolling time, and as a result the layered particle orientation is not well

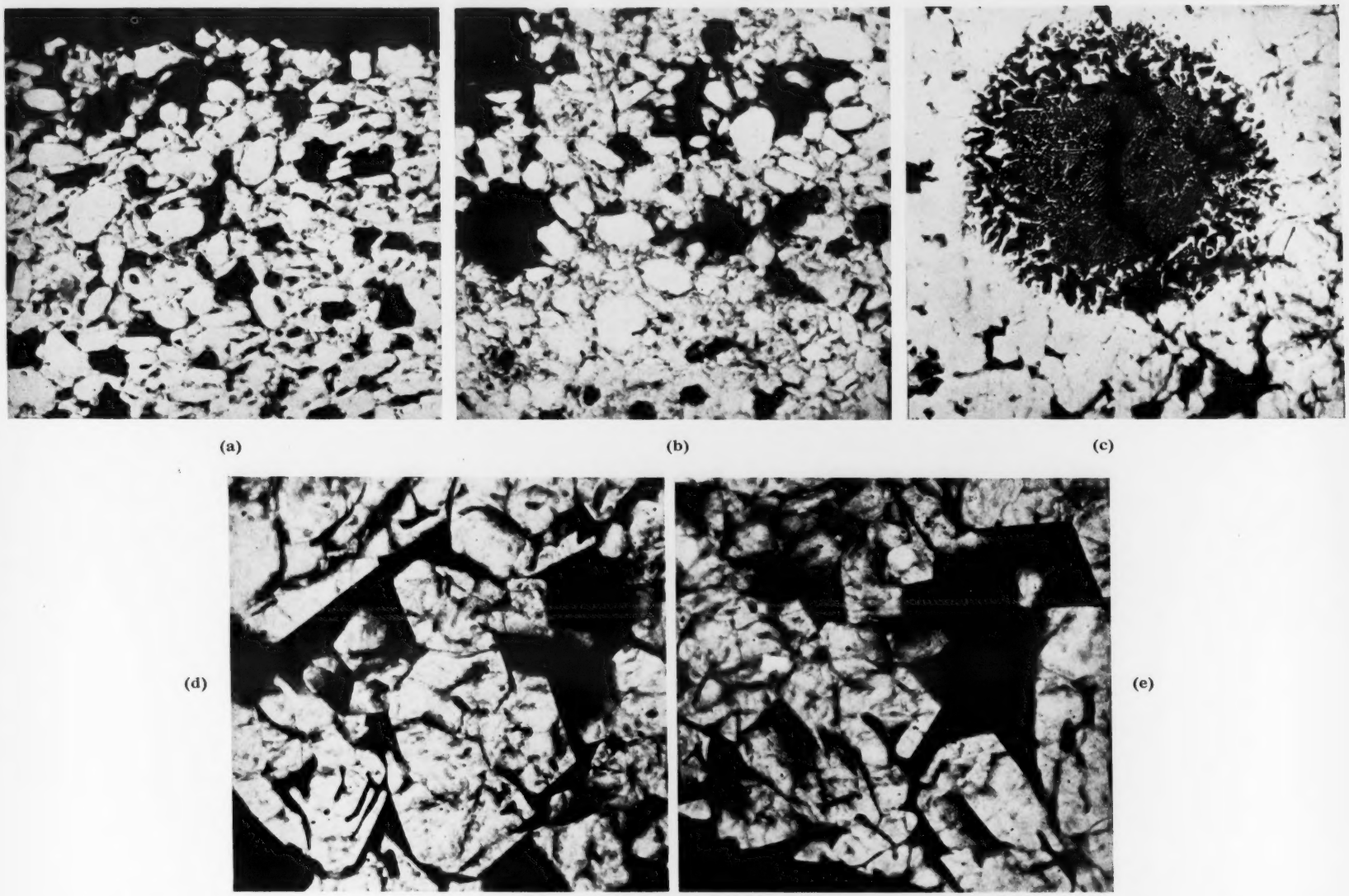


(a) (b)



(c)

Fig. 9—Gas-fired pellets of Sierra Leone concentrates: (*a*) Fully oxidized; (*b*) reduced to magnetite; (*c*) coarse magnetite and secondary dendrites in slag × 65



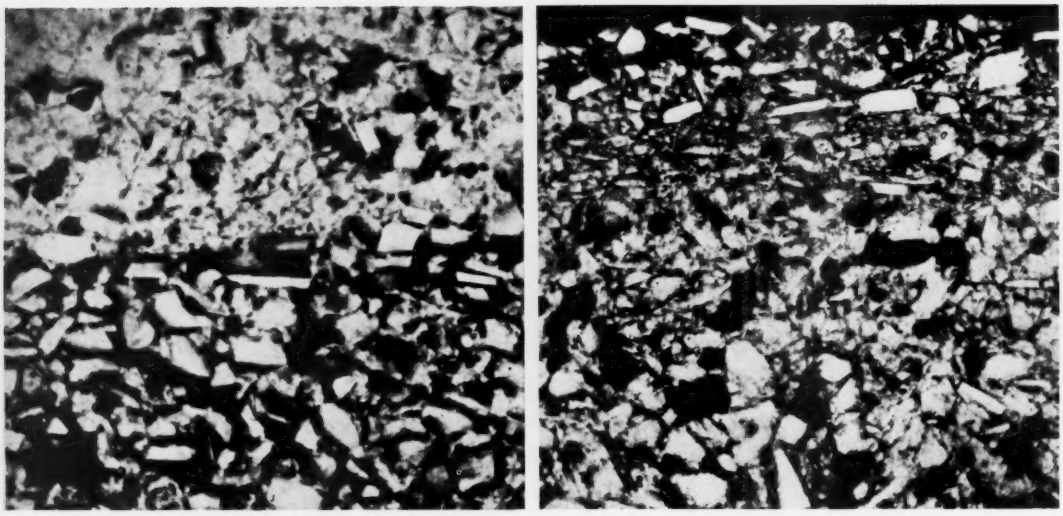
(a) (b) (c)

(d) (e)

Fig. 10—Coke-fired pellets of Sierra Leone concentrates: (*a*) and (*b*) Edge of pellet and oxidized outer zone; (*c*) magnetite and slag pool with dendrites in the inner zone; (*d*) and (*e*) coarse magnetite and fayalite near the central cavity × 65

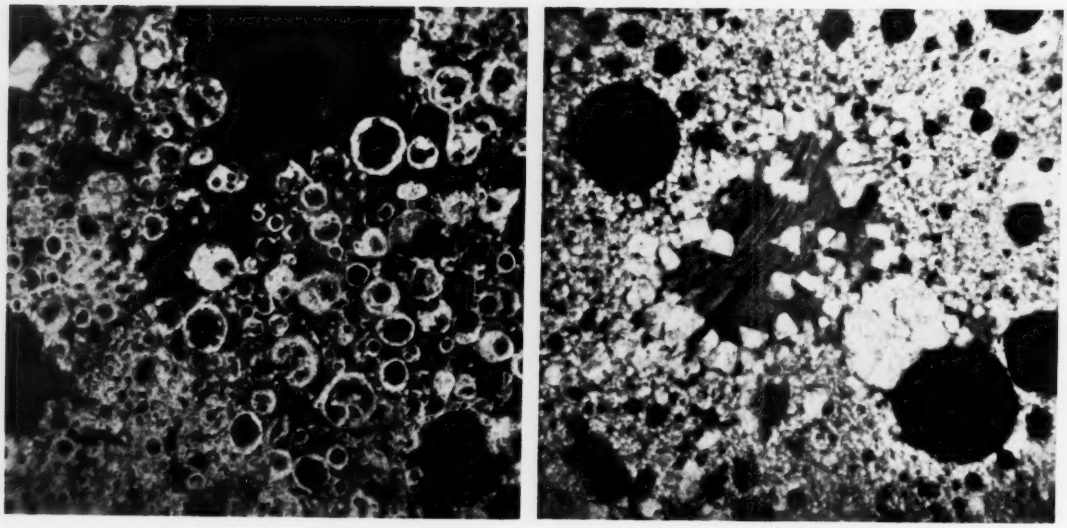
Fig. 10—Coke-fired pellets of Sierra Leone concentrates: (*a*) and (*b*) Edge of pellet and oxidized outer zone; (*c*) magnetite and slag pool with dendrites in the inner zone; (*d*) and (*e*) coarse magnetite and fayalite near the central cavity × 65

Fig. 11—Enlarged photograph of cavity in overfired pellet, showing magnetite crystals × approx. 10



(a) (b)

Fig. 12—Textures of gas-fired pellets: (*a*) Effect of different size gradings in the feed; (*b*) a layered skin on unorientated material produced by finish-rolling after rapid build-up × 65



(a) (b)

Fig. 13—Pellets of flash-roasted pyrites residues: (*a*) Cenospheres well preserved in a gas-fired pellet; (*b*) fayalite pool and magnetite in a coke-fired pellet × 65

developed. It has not so far been established whether an orderly layered alignment of particles is more advantageous than a disordered texture. Neither crushing nor permeability tests give a clear indication in favour of either.

A well-layered pellet skin has the advantage of being less susceptible to abrasion, whereas rapid pellet build-up obviously increases the capacity of the plant. In practice both are achieved by using a fairly long rolling drum, with the last third of its length serving merely for a final rolling without further build-up. Figure 12*b* shows a pellet produced under such conditions. The outer skin, near the upper margin of the figure, has a marked tangential alignment of the coarse particles, whilst the main body of the pellet shows a marked absence of preferential orientation. This pellet is typical of the good average product obtained from the pilot plant with external gas firing.

Work on Sierra Leone hematite having been completed, attention was directed to pelletizing other materials, including various Scandinavian magnetite concentrates and flash-roasted pyrites residues. The latter yielded results of some interest but before passing on to these, it may be useful to consider briefly two papers[6, 7] dealing with American work on micro-studies of pellets.

The American work was carried out under conditions differing from those described in this paper in three important aspects:

(i) The iron-bearing constituent of the concentrate was chiefly magnetite
(ii) Slagging gangue constituents amounted to 10–12%, compared with 4–5% in Sierra Leone hematite concentrates
(iii) The magnetite concentrates were of much finer grain, about 90% being below 200 mesh and more than 60% being below 24 μ, compared with 60% below 200 mesh and about 30% below 24 μ in the case of the hematite concentrate.

With magnetite pellets, the whole of the firing process is characterized by reactions which begin to occur at low temperatures, as pointed out by the American authors. Hematite, however, remains virtually inert up to about 1250° C., provided that oxidizing conditions are maintained. No bonding mechanism is therefore available until hematite diffusion begins, making it advantageous to employ some other bonding medium to maintain strength over the temperature range from about 100° to 1250° C. The American authors found a gradual rise in the crushing strength of their pellets due to various iron-oxide reactions and slag formation. In the case of Sierra Leone hematite pellets, series of tests with pellets fired to various temperatures showed that, after drying, virtually no strength was evident until a sharp rise in strength occurred between 1250° and 1300° C., maximum strength being reached at the latter temperature. As stated above, sodium carbonate has been selected as the most efficient source of bonding forces for dry hematite pellets. Cooke and Stowasser[7] mention sodium carbonate in a list of other inorganic additives considered by them, but they do not further comment on its performance and seem to have rejected it together with all other additives as ineffective or harmful with their material.

Apart from the differences in behaviour between magnetite and hematite, the contrast in slag content, or more specifically in slag volume, must be considered. About 10–12 wt.-% of slag of about 2·7 sp.gr. corresponds to 20–25% of the pellet volume in the case of the American magnetite pellets. Sierra Leone hematite pellets contain a maximum of about 4–5% of slagging constituents, 3·8% being a common average figure. This would result in an average of 7½% slag volume, with a maximum of 10%.

This small volume of slag does not contribute sufficiently to pellet strength; pellets fired to 1280° C. showed crushing strengths of a few hundred pounds, whereas pellets fired to 1300° C. exceeded 1500 lb. in strength. With magnetite pellets the high initial volume of slag is increased further by relatively early solution of a large proportion of iron; the slag is probably the important bonding agent in magnetite pellets both during firing and after the maximum temperature has been reached. It is not surprising that an oxidized outer skin was found to be very beneficial in isolating the slagged bulk of each pellet from its surroundings, a condition corresponding exactly to the present authors' findings when coke was incorporated in the hematite pellets. Combustion of the coke inside the pellets caused reduction of hematite to magnetite, and the latter reacted with the slag constituents from ore and coke to give an iron-rich slag. With an ample supply of air streaming through the kiln, oxidizing conditions were usually maintained at the surface of each pellet, preserving an outer layer of inert hematite. If bad air distribution occurred as a result of dust blanketing in part of the charge, the outer hematite shells were lost and pellets coagulated into partly molten masses of coarsely crystalline magnetite and fayalite slag.

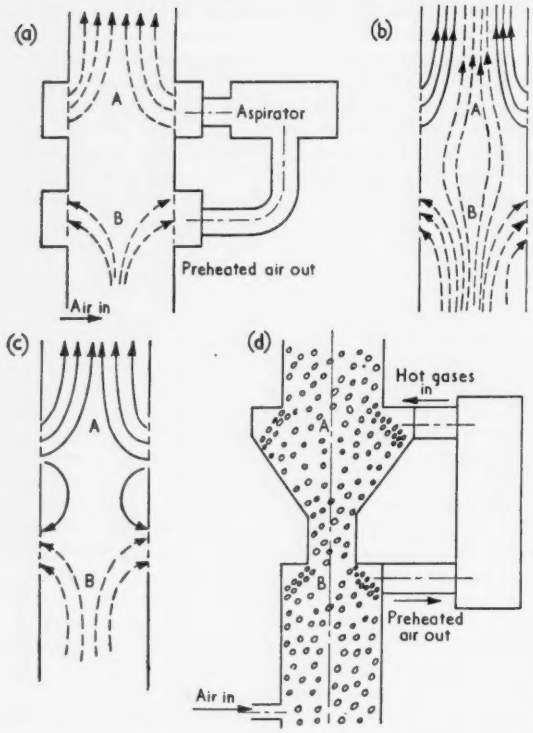
The formation of much slag should always be regarded as detrimental from the point of view of porosity and reducibility of the pellets. The contrast between the high porosity of unreduced hematite pellets (gas-fired) and partly reduced coke-fired pellets is striking enough and a drop in reducibility is to be expected, even if the formation of fayalite is not taken into account.

Reducibility tests were carried out in hydrogen at 750° C. to compare various commercial ores with Sierra Leone pellets. Results were as follows:

Ore	Reduction after 1 hr., %
Kiruna	80
Itabera	86
Ouenza	97
Sierra Leone pellet (gas-fired, fully oxidized)	94
Sierra Leone pellet (coke-fired, partly reduced, and slagged)	82

Reducibility of an unaltered hematite pellet is thus approaching that of the most easily reducible ores, and slagged magnetite pellets are nearly as bad as the most irreducible ore.

There is, in addition, the consideration of purely physical strength. Slag formation and growth of magnetite prevent the hematite diffusion bonding without replacing the latter's high strength value. The present authors agree, however, with the American authors that a little slag bonding in addition to



(a) $p_A = p_B$ (b) $p_A < p_B$ (c) $p_A > p_B$

Fig. 14—Diagram to illustrate gas flows with recuperation

hematite diffusion may be a desirable improvement. The addition of sodium carbonate is probably of marked advantage in this respect, provided that the proportion of slagging constituents is quite small. In spite of the lack of proof, it is believed that sodium carbonate imparts a greatly increased mobility to the slag in Sierra Leone pellets, so that the slag may permeate the pellet fabric without interfering with the hematite bond. Instead of remaining in the form of isolated and ineffective particles, the slag thus forms a system of skins on the free surfaces of the hematite, not reducing the overall porosity but increasing the overall strength of the pellets. The addition of sodium carbonate raised the strength of fired pellets from about 1500 lb. to about 3000 lb., and, with higher concentrations of sodium carbonate, crushing strengths above 6000 lb. were obtained.

The relatively low crushing strength of American magnetite pellets (a maximum of 250 kg. has been quoted[6]) is somewhat unexpected in view of the very fine grain size of the material used. The fine size does seem to result in good green strength, because a crushing strength of 1·2 kg. (2·64 lb.) is quoted for air-dried pellets, a high figure considering that no binder is used.

Pelletizing of flash-roasted pyrites residues was found to conform with previous experience, but additional care was desirable in maintaining oxidizing conditions during firing so as to minimize residual sulphur.

The essential iron-bearing constituent of the pyrites residues was found to be hematite, but the flash-roasting process had introduced a curious structural feature. Microscopic studies of thin sections and radiographs show that the material consists very largely of hollow hematite spheres ('cenospheres') which occur as free individual particles ranging in diameter from 0·01 to 0·40 mm., 0·07–0·10 mm. being most common. The larger spheres frequently enclose two or three smaller ones, resulting in a peculiar cellular appearance. Occasional angular grains of quartz are the only other notable constituent of the pyrites residues.

It was found that the cenospheres provide a simple guide for speedy assessment of the effectiveness of any firing trial. Good preservation of the cenosphere structure (Fig. 13*a*) always indicates strong well-fired pellets, a result notably achieved with external gas firing under oxidizing conditions.

Pellets fired by means of incorporated coke always failed to retain complete oxidation. Their cores were generally reduced to magnetite, accompanied by the formation of iron-rich slag, loss of porosity, and crystallization of fayalite (Fig. 13*b*). It was generally possible to maintain an outer oxidized layer with well-preserved cenospheres. The latter indicate that the outer layer remained oxidized throughout the firing and was not first reduced and later re-oxidized during cooling. Had reduction to magnetite occurred, this would have been accompanied by reaction with silica and almost certain destruction of the cenosphere structure.

Coherence between the reduced slagged core and the unaltered outer layer is mostly weak and in many pellets the core can be seen to have shrunk from the skin over considerable areas. When the porosity of the coke-fired pellets is lowered, they assume a cavernous structure, presumably owing to gases evolved by the burning coke. All these factors result in a lower crushing strength compared with gas-fired unreduced pellets.

HEAT BALANCES

The establishment of a detailed thermal balance for an industrial process requires some effort to obtain reliable numerical data which are self-consistent, and the conditions of operations of the pilot-plant kiln make the problem difficult. Heat losses from the shaft are high and include effects of cracks and other defects in the structure, for which it is difficult to make due allowance, and observations on flow rates, temperatures, etc., have to be made as opportunities allow by the staff engaged in the operation. The individual heat-loss figures suggested for gas-firing conditions are therefore not of high accuracy, but their mutual consistency gives reason to hope that the overall picture presented is not seriously in error. Study of the fired pellets in the light of laboratory firing trials gives a useful estimate of the maximum temperature reached, and this can be compared with an estimate based on gas temperature and mass flow on the lines developed in Appendix II. This makes it possible to calculate the heat balance in two stages, relating to the heating of the charge to firing temperature and the dissipation of heat in the lower portion of the

furnace. It is then possible to determine the effect on overall efficiency of reducing the heat losses of the first stage. Second-stage losses merely affect the discharge temperature of the pellets and have no influence on total thermal requirements. This last statement would no longer be true if the sensible heat of the product could be used to preheat the combustion air. Some American workers have proposed designs to allow this to be done. The problem of using the lower half of the shaft as a recuperator seems to be beset with difficulties, as it is important to avoid passage of air from the recuperation zone to the firing zone or of combustion gases in the reverse direction. As Fig. 14 shows, the pressures at A and B must be kept equal, and this can only be done by control of air input and aspiration rate. If $p_A < p_B$, air travelling up the shaft will deflect the combustion-gas stream away from the axis and result in underfiring of the pellets in this region. With $p_A > p_B$, some combustion gases join the air circulation and, although the total heat in the system is unaffected, the heat available above the critical firing temperature is reduced, with consequent loss in overall efficiency. As the preheated air can only be extracted at level B by some form of gas or air aspirator, the control of p_B is a major problem. The American proposal is to minimize the effect of pressure differentials by imposing a restriction to gas flow, as in Fig. 14*d*. Experience on the materials used at Redcar suggests that free descent of the charge would be impossible with such a design and, furthermore, that recuperation in the kiln shaft is not a practical proposition.

Reverting to the heat balance ascertained for gas-firing conditions, the following data relate to a trial using Sierra Leone concentrates referred to in Fig. 8*a*:

Coke-oven-gas consumption	1680 cu. ft./hr.
Net calorific value	463 B.Th.U./cu. ft. (sat. 60° F.)
Air for complete combustion	7600 cu. ft./hr.
Total air to burner	12,360 ,, ,,
Auxiliary air supply (for cooling)	3000 ,, ,,
Dry weight of pellets	840 lb./hr.
Associated water	73 ,,
Combustion-chamber temp. (observed)	1450° C.
Theoretical flame temp.	1477° C.
Top gas temp. (observed)	150° C.
Final pellet discharge temp. (estimated)	600° C.

Balance for Heating Section (values in B.Th.U./hr.)

Heat Input:	
From combustion of coke-oven gas	777,500
Sensible heat of gas and air at 15° C.	7400
	784,900
Heat Consumed:	
To pellets 15–1350° C.	429,900
To associated water 15° (liq.) to 150° C. (vap.)	85,250
Radiation and convection losses	33,300
Auxiliary water-cooling	91,200
Top gas at 150° C. (89·25% of total combustion products)	62,200
Gases descending stack at flame temp. (10·75% of total volume)	83,050
	784,900

Balance for Cooling Section (values in B.Th.U./hr.)

Heat Input:	
Heat content of pellets at 1350° C.	433,900
Gases from upper section	83,050
Sensible heat of auxiliary air	1500
	518,450
Heat Consumed:	
Pellets discharged at 600° C.	184,500
Air and combustion gases at 600° C.	97,050
Radiation and cooling losses (bricked section)	20,800
Water-cooling	155,500
Radiation and cooling losses (unlined discharge section)	60,600
	518,450

Perhaps the most striking feature of these figures is the excessive heat losses in the upper section and, in particular, the losses by water-cooling. This relates to a series of hot spots, the main one being around the burner entry. With the mistaken intention of providing ready access to the whole burner assembly for possible modification, the burner assembly was made detachable, and this meant that slight distortion of the supporting plate allowed the combustion gases to leak in and circulate behind it. If the kiln were rebuilt, this weakness could be eliminated and the total shell losses of the upper section could be reduced to less than 30,000 B.Th.U./hr.

The other conspicuous loss in the upper section is that shown as 'Gases descending stack at flame temp.' This can be prevented by adequate sealing at the discharge point. With these two changes, the vital balance of the heating-up region would become:

Heat Input:	
From combustion of coke-oven gas	593,250
Sensible heat of gas and air at 15° C.	5400
	598,650
Heat Consumed:	
To pellets 15–1350° C.	429,900
To associated water 15–150° C.	85,250
Radiation and convection losses	30,000
Top gas at 150° C.	53,500
	598,650

For this the gas requirement is 1280 cu. ft./hr. = 5·93 therms/hr. = 3410 cu. ft./ton = 15·8 therms/ton. There seems to be no reason why a commercial plant should not attain or improve on these figures, as the external heat losses per ton will decline with increase in size of plant.

Under conditions of solid-fuel firing, the heat balance is much less informative, as a large proportion of the total heat requirement is derived by recuperation. Any theoretical check on the observed data would involve a detailed study of heat exchange in the lower shaft and of the diffusion of air into and CO_2 out of the pellets themselves. Too little is known of the mechanism of the latter process, or even of the temperature distribution in the thin firing zone, to permit any reliable deductions to be made.

It has therefore only been found possible to present typical overall heat balances for two contrasting trial runs on Sierra Leone and Sydvaranger concentrates,

respectively. In the latter case the heat of oxidation of Fe_3O_4 makes an important contribution to the economy of the process, although in the trial chosen the conversion was only half-completed. By contrast, some reduction of the Sierra Leone concentrates occurred with a corresponding thermal demand. In the second case, the firing zone was maintained nearer the stockline, which has resulted in a higher top gas loss and a lower figure for external loss. In both trials the table discharge was unenclosed, and it has been assumed that the air entering near the bottom of the shaft escaped at this point.

	Sierra Leone Concentrates	Sydvaranger Concentrates
Coke used, %	3·6	2·0
,, ,, lb./hr.	36	20
Dry weight of pellets, lb./hr.	1000	1000
Associated water, lb./hr.	87	69
Air supply, cu. ft./hr.	13,600	13,700
Auxiliary air supply, cu. ft./hr.	5000	5000
Top gas temp., ° C.	100	250
Final pellet discharge temp. (estimated), ° C.	300	150
Reduction, %	38	—
Oxidation, %	—	47
Heat Input, B.Th.U./hr.:		
Combustion of coke	460,080	255,600
Oxidation of magnetite	—	108,700
	460,080	364,300
Heat Consumed, B.Th.U./hr.:		
Reduction of hematite	85,000	—
Net heat to pellets	92,300	43,700
To associated water	97,800	86,400
To air and combustion products	92,180	132,600
Difference (radiation, convection, etc.)	92,800	101,600
	460,080	364,300

The external losses are high in both cases owing to poor recuperation arising from the introduction of air through the original ports rather than lower down the stack. The heat not recovered by recuperation must, of course, appear as sensible heat of the product and cooling losses from the lower portion of the shaft.

ECONOMIC CONSIDERATIONS

As a natural consequence of the solution of the technical problems involved in pelletizing, it was necessary to forecast the probable costs which would be involved on a commercial scale. With experience limited to operations and plant at a level of ½ ton/hr., only approximate figures can be obtained.

The contributory elements to the cost may be examined separately as follows:

(i) Capital cost of plant

(ii) Grinding cost in the case of Sierra Leone concentrates (this does not apply to magnetite concentrates or flash-roasted pyrites)

(iii) Cost of balling operation
- (*a*) Labour
- (*b*) Power
- (*c*) Binder
- (*d*) Maintenance

(iv) Cost of firing
- (*a*) Fuel
- (*b*) Labour
- (*c*) Refractories.

Capital Costs

As full details and drawings of a commercial-scale plant have not yet been worked out, considerable difficulty was naturally experienced in arriving at an estimate of cost. The method adopted was to draw up a schedule of the more important items of plant in varying degrees of detail, together with a schematic layout plan to show the order of size of the building and facilities required. These were submitted to an ironworks operator with extensive experience of plant construction, who undertook the difficult task of estimating an overall cost figure.

The basis of design was the use of a mixture of Sierra Leone concentrates (to be ground) and fines with pulverized coal or coke added for the firing process. The output chosen was 30 tons/hr., *i.e.*, about 250,000 tons/year.

For the preparation stage, allowance was made for a ball mill 8 ft. × 6 ft. with a 42-in. classifier in circuit to deal with 15 tons/hr. of concentrates. Settling tanks fitted with vibrators were included (*see* Appendix I). For the combined materials to be pelletized, allowance was made for two twin-shaft pug mills, each capable of treating the whole feed. The balling stage was designed for three drum units (one spare), each 7 ft. dia. × 15 ft. long, with suitable provision for screening and transport of returns. Transport of the product to the kilns was to be by a specially designed conveyor with facilities to distribute the pellets along the stockline (*e.g.*, a travelling tripper). Two rectangular kilns were included, each 6 ft. × 16 ft. in cross-section.

The estimated cost (1952), including buildings and foundations but excluding access facilities and provision of works services, was £200,000. Without more detailed examination the capital cost can only be estimated as about £1 per ton per year, a figure of similar order to that applicable to sintering plants.

Grinding

In the wet milling trials referred to in Appendix I a power consumption of 12½ kWh./ton was observed. The cost of this would need to be supplemented by a figure for wear of balls and liners, which could only be obtained with accuracy in a long-term trial. Our limited experience suggests a ball consumption of 1 lb./ton of output. Provided that a suitable balance could be maintained between ground concentrates and other relatively dry fines or concentrates, the cost of the settling and partial dewatering arrangements envisaged would amount to a small fraction of the total grinding cost.

Balling and Firing

In costing a particular scheme, some assessment has to be made of the most probable binder addition, based on pilot-plant trials on the ores to be used; the maximum proportion of sodium carbonate used hitherto has been 0·3%. Power requirements for this stage have been worked out from the probable horsepower of motors for fans and conveyors, and found to amount to 2–3 kWh./ton. Labour requirements can be estimated in relation to the duties to be performed: a total shift labour force of six seems adequate for a full-scale plant, excluding stockyard and disposal

services. Maintenance costs are more difficult to forecast, but as nearly all the equipment works at normal temperatures, they are likely to be appreciably lower than on sinter plants.

The fuel to be used will depend on local conditions to a marked degree. With coke, experiments have shown a requirement of about 3% by weight. This coke has been reduced to a size grading similar to the ore, which would seem essential to ensure adequate dispersion in the green pellets. Coke breeze, although relatively cheap, is abrasive and expensive to grind, so that it is perhaps wiser to allow for pulverized coal. The cost of this would depend on the possibilities of using low-grade material, such as washery slurry. Making allowance for the difference in analysis between coal and coke, the fuel requirement might be 4–5%.

External firing may be preferable in certain cases. Coke-oven gas has been used in all the experimental gas firing to date, the anticipated requirement being 3500 cu. ft./ton. Blast-furnace gas has been considered, but this would require efficient preheating of the air by recuperation from the fired pellets. The control of flow of air to avoid interference with the use of blast-furnace gas, except in mixtures with coke-oven gas, is hardly realistic at the present stage of development. Oil firing might have advantages in convenience and ease of control; the requirement would be about 9 gal./ton.

Total Cost

The costs of operating the process will depend so largely on local conditions and on the raw materials to be used that it is not possible to arrive at a figure which is generally applicable. However, in certain specific cases calculation has led to the conclusion that costs will be somewhat less than in current sintering practice.

CONCLUSIONS

The work outlined in this paper has shown that both hematite and magnetite concentrates can be successfully pelletized on a pilot-plant scale. The translation of the process to a commercial scale may well bring to light further problems peculiar to larger units, but the experience already gained not only provides a basis for development, but also gives a guide to the general economic prospects of the process.

The possibilities of extending work on pelletizing to a commercial scale in the U.K. are still under discussion, and it may not be out of place to review certain relevant economic considerations. The problem of processing imported concentrates in the U.K. is very different from that facing U.S. and Swedish producers. Current imports of concentrates are only of the order of 500,000 tons/year, although circumstances might arise in which this figure would increase. With a total sintering capacity of 4½ million tons and a prospective increase to 8 million tons by 1955, these concentrates can be agglomerated by sintering either alone or mixed with screenings of other ores. High proportions of concentrates result in a decrease in sinter output and a consequent increase in cost, although reasonable performance figures can be maintained. The present authors' calculations suggest that pelletizing material of this character in an installation producing about 250,000 tons/year may prove to be more economical than sintering, but that the cost differential will not be large. The observed superiority of pellets over sinter in regard to porosity and degree of oxidation suggests that improved blast-furnace efficiency may well be a vital factor in encouraging the adoption of the process, but in the absence of operating experience it is not possible to make due allowance for this. In the present state of development of ore preparation in the U.K. it seems appropriate to consider the establishment of one suitably located pelletizing plant to deal with the bulk of the present imports rather than to supplement sintering facilities in a number of works with less economic units. An examination of the commercial arguments bearing on such a project and of the most suitable location is outside the scope of this paper.

Flash-roasted pyrites residues present a different picture. The material arises in a finer state of subdivision than Sierra Leone concentrates, for example, and additions to conventional sinter-plant burdens must be limited to a small percentage to avoid an adverse effect on sinter-bed permeability and sinter output. The producers are in general not favourably located in relation to blast-furnace plants, and the output of individual plants is only about 20,000–30,000 tons/year. Assembly of all these quantities at a central pelletizing plant seems to be inappropriate for several reasons:

(i) The presence of copper and other non-ferrous elements in the residues makes wide dispersion among blast-furnace plants desirable

(ii) Thus transport charges would be incurred on both raw material and product

(iii) Such fine material cannot be transported dry and would need to be pugged and prevented from drying out

(iv) Thus the raw material would be of unknown and variable moisture content; control of water addition for pelletizing is easier with an initially dry material.

Pelletizing at source of quantities of 100 tons/day would be more costly than on a larger scale, and the cost might also be increased by the need to use oil fuel, but the product would be convenient for transport and dispersion amongst the blast-furnace plants. The experimental work already carried out shows that this material can be satisfactorily pelletized, and the development of plants of a suitable scale is worthy of serious consideration.

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The work has involved the co-operation of two teams of investigators, at the Blast Furnace Research Laboratory, Imperial College, and at the B.I.S.R.A. North-East Coast Laboratories, and such success as

has been achieved has been due to the wholehearted co-operation of the members, both in ideas and in hard work. The teams have included the following: Mrs. R. M. Hodges, Mr. J. Docherty, Mr. B. S. Ekelund, Mr. E. Greenwell, Mr. W. E. Jones, Mr. P. R. Sirs, Mr. G. E. Turner, and Mr. J. F. Whone.

Finally, the authors wish to thank Dorman Long and Co., Ltd., for their generosity in providing accommodation and technical facilities.

APPENDIX I

Note on Grinding Trials on Sierra Leone Concentrates

Sierra Leone concentrates are too coarse for pelletizing as-received and it was necessary to make provision for grinding to a suitable size. Tests were therefore arranged on several mills of different types and an assessment was made of throughput and power consumption. Air-swept ball mills and a ring roller mill were unsatisfactory in both respects, but more encouraging results were obtained with an Atritor dry grinding mill and with wet ball milling.

The Atritor was chosen for the pilot plant on the grounds of early delivery. It is a high-speed mill consisting of a rotating disc carrying rows of pegs which pass with small clearance between fixed rows of pegs. The mill is swept with hot air which serves to dry the feed and the product is collected in a cyclone. The size grading of the product can be varied by suitable adjustment, giving a range of -200 mesh fraction from 40% to 90%. Power requirements are of the order of 15 kWh./ton when grinding to 60% below 200 mesh. The mill proved extremely valuable in providing an adequate supply of dry ore of appropriate size grading for the test plant. As was to be expected, it is not suitable for commercial use with these highly abrasive concentrates because of excessive wear of the grinding elements. Hard facing of the pegs with stellite, tungsten carbide, etc., improved the life, but maintenance costs were still quite unacceptable. At a later stage, therefore, grinding experiments were continued with a 3 ft. $\times$ 10 in. Hardinge ball mill, working in closed circuit with a spiral classifier or a Driessen hydraulic cyclone. With a charge of 1120 lb. of $2\frac{1}{2}$–$1\frac{1}{2}$-in. balls, a throughput of 8 cwt./hr. could be maintained. Under these conditions the overflow product showed 70% below 200 mesh with 7% above 100 mesh and the power consumption averaged $12\frac{1}{2}$ kWh./ton. These figures are encouraging, provided that economical means can be found for dewatering the product for pelletizing.

A material with the optimum moisture for pelletizing is not visibly wet and cannot be made to flow like a liquid suspension. Ground Sierra Leone concentrates, which pelletize at 9% of water, may be made to flow under conditions of vibration at, say, 11% and form a viscous suspension which can be pumped at about 20% of water. In this latter case, the volume percentage of solids is only about 45, as the density of the solid is about 5. Starting with a pulp density from the classifier overflow of about 45 wt.-% of solids, it is possible to effect a considerable degree of dewatering by hydraulic cyclones. Experiments on these lines resulted in a reduction of water content to about 21%. Simple settling over periods of a few minutes will give a sediment with about 16% of water, and if the settling tank is vibrated for, say, 5 min. this figure can be reduced to 13%. Solids recovery is complete.

Some tests were also carried out with a continuous high-speed centrifuge and with vacuum drum filters, but it seemed likely that power costs would be excessive. Moreover, in none of these tests was the moisture content of the product as low as the 13% mentioned above.

It was therefore concluded that the most promising line was to use vibration-assisted settling of a pulp previously concentrated by cyclone, and thereafter to mix in suitable dry ore fines to reach a suitable moisture content for balling. Certain mixtures of ground concentrates and fines examined demand a moisture content of 10–15% for pelletizing, which is very easy to achieve. If the addition of dry fines is inconvenient or undesirable, it would seem to be necessary to resort to partial drying for the final stages of water removal.

APPENDIX II

Heat-Transfer Conditions in Pellet Firing

By L. F. Daws and H. Herne

The Shaft Furnace as a Heat Exchanger

The process of heating the pellets in the furnace is a counterflow heat-exchange process. There are two obstructions to heat flow:

(i) The finite conductivity of the pellets
(ii) The finite heat-transfer coefficient across the boundary layer between the heating medium in the form of gas, and the pellets.

The second of these largely determines the time a pellet must be in contact with the heating medium, but the first, although affecting this to some extent, is of more importance in that it limits the degree of temperature uniformity throughout the pellet.

If it can be shown that obstruction due to the first cause is not serious in comparison with that due to the second, it will be possible to treat the counterflow heat-exchange process much more simply theoretically. A study of these two obstructions to heat flow is also desirable from the practical point of view to give quantitative design data.

Relative Importance of Conduction in the Particle and Heat Transfer from Gas to Solid

Saunders and Ford[8] give a criterion whereby if

$$\frac{V\rho sd}{K} < 4 \qquad \text{.....(1)}$$

the conductivity of the ore may be neglected, where

V = mean linear velocity of gas entering bed
s = specific heat per unit mass of gas
ρ = density of gas
K = thermal conductivity of pellets.

In the process Firth[1] describes, gas velocities of the order of 500 cm./sec. are apparently obtainable. For Fe_2O_3 from Sierra Leone concentrates $K = 10^{-3}$ cal./cm./sec./° C., and if it is assumed that the heating medium in the pelletizing chamber is burnt blast-furnace gas plus 25% excess air,

$$\rho s = 0{\cdot}329 \times 10^{-3} \text{ cal./c.c./° C.},$$

so that, for $d = 1{\cdot}27$ cm., from equation (1),

$$V < 100 \text{ cm./sec.},$$

if conduction in the pellet is to be neglected.

It is necessary, therefore, to study this question more closely to see how important conduction is in relation to convection.

Heat Flow in Sphere with Heat Transfer at the Surface from a Medium, the Temperature of which Varies Linearly with Time

List of Symbols

θ = Temperature at distance r from the centre of a pellet at time t
K = Thermal conductivity of the material of the pellet
k = Thermal diffusivity = $K/\rho s$, where ρ and s are respectively the density and the specific heat per unit mass of the pellet material
a = Radius of pellet
α = Heat-transfer coefficient from gas to pellet or gas conductance
θ_g = Gas temperature = $\theta_0 + ct$, where θ_0 is the outgoing gas temperature and c is a constant.

In this study it is assumed for simplicity that the temperature of the gases in the pelletizing chamber varies in such a way that in relation to a descending pellet of Fe_2O_3 it appears to change linearly with time. The heat flow equations to be solved are:

$$\frac{\partial\theta}{\partial t} = k\left(\frac{\partial\theta^2}{\partial r^2} + \frac{2}{r}\frac{\partial\theta}{\partial r}\right),\ 0 < r < a \quad \ldots\ldots(2)$$

$$\theta(r, 0) = 0 \quad \ldots\ldots(3)$$

$$K\frac{\partial\theta}{\partial r} + \alpha(\theta - \theta_g) = 0,\ r = a \quad \ldots\ldots(4)$$

It can be shown that at the centre of a pellet (*i.e.*, at $r = 0$) the temperature must take the form

$$\theta = \theta_0 + ct - 2ha\theta_0 \sum_{n=1}^{\infty} e^{-k\beta_n^2 t} \frac{[\alpha^2\beta_n^2 + (ah-1)^2]^{\frac{1}{2}}}{[\alpha^2\beta_n^2 + ah(ah-1)]} - \frac{2\rho s c h a^3}{K} \sum_{n=1}^{\infty} \left(1 - e^{-k\beta_n^2 t}\right) \frac{[\alpha^2\beta_n^2 + (ah-1)^2]^{\frac{1}{2}}}{\alpha^2\beta_n^2[\alpha^2\beta_n^2 + ah(ah-1)]} \quad (5)$$

where β_n are the real positive roots of

$$\alpha\beta_n \cot\alpha\beta_n + \frac{a\alpha}{K} - 1 = 0 \quad \ldots\ldots(6)$$

and where $h = \alpha/K$, $k = K/\rho s$.

For a particle having infinite conductivity, the rate of heat transfer at the surface is given by

$$\frac{4}{3}\pi a^3 \rho s \frac{\partial\theta}{\partial t} = 4\pi a^2 \alpha(\theta_0 + ct - \theta)$$

$$i.e.,\ \frac{\partial\theta}{\partial t} + \frac{3\alpha}{a\rho s}\theta = \frac{3}{a\rho s}(\theta_0 + ct) \quad \ldots\ldots(7)$$

The temperature of a pellet with infinite conductivity is therefore given by the solution of this equation, obeying the boundary condition as in equation (5) of

$$\theta(r, 0) = 0$$

$$i.e.,\quad \theta = ct + \left(\theta_0 - \frac{a\rho s c}{3\alpha}\right)\left(1 - \exp\left\{\frac{-3\alpha}{a\rho s}t\right\}\right) \quad \ldots\ldots(8)$$

The difference in temperatures $\Delta\theta$ at the centre of a pellet, because of its finite conductivity (*i.e.*, the ratio of heat conduction in the skin to that in the body of the pellet), is found by subtracting equation (5) from equation (8).

It is known from static experiments that pellets of Sierra Leone concentrates harden at about 1350° C. Furthermore, an inlet gas temperature of 1450° C. can reasonably be obtained by burning cold blast-furnace gas with air preheated to 540° C. The air flow is taken to be the theoretical amount required for complete combustion plus 25%. This ensures that the atmosphere in the chamber is oxidizing.

Under these conditions it will be possible to harden the pellets, provided that the sum of the temperature lag between pellets and gas due to the finite conductivity of the pellets plus that due to the finite heat-transfer coefficient between gas and pellets is less than 100° C. when the pellets leave the furnace, and also that the temperature gradient in the pellets is not affected to any great extent by either of these values.

If the cross-sectional area of the pelletizing chamber is 2·2 sq. ft., corresponding to a chamber dia. of 20 in., the rate of throughput of pellets must be about 1000 lb./hr. for the rate per unit area of cross-section to be of the same order as in Firth's process. The heating time of the pellets will then be about 3000 sec. Then, taking $\theta_0 = 200°$ C. for reasonable efficiency, c in the expression for the gas temperature $\theta_0 = ct$ is given by

$$c = \frac{1450 - 200}{3000} = 0{\cdot}42.$$

Using this value for c, the expression for $\Delta\theta$ has been computed for values of ah from 0·2 to 2·0 and for times for which the exponential terms in equation (5) are less than 0·05 θ_0, 0·02 θ_0, and 0, respectively. The corresponding temperature lags due to finite heat-transfer coefficients

$$\Delta\theta_1 = \frac{a\rho s c}{3\alpha} + \left(\theta_0 - \frac{a\rho s c}{3\alpha}\right)\exp\left\{\frac{-3\alpha}{a\rho s}t\right\} \quad \ldots\ldots(9)$$

have also been computed. The calculations have been carried out for a pellet size of $\frac{1}{2}$ in.

The values for $\Delta\theta$ and $\Delta\theta_1$ are given in Table I. It can be seen that $\Delta\theta$ is everywhere less than 35° C., so that the obstruction to heat flow due to the finite conductivity of the pellets is not a serious one. The temperature lag due to finite heat-transfer coefficients is quite large for the smaller values of ah, so that heat-transfer coefficients of less than $0{\cdot}8 \times 10^{-3}$ cal./sq. cm./sec./° C. will cause a much more serious obstruction to heat transfer than the finite conductivity of the pellets. For values of ah greater than 0·5 (*i.e.*, for heat-transfer coefficients greater than $0{\cdot}8 \times 10^{-3}$ cal./sq. cm./sec.) the sum of the temperature lags is less than 100° C.

Optimum Conditions

Consider the process of pelletization taking place

Table I

COMPARATIVE TEMPERATURE LAGS FOR FINITE CONDUCTIVITY AND HEAT-TRANSFER COEFFICIENTS

ah	$10a^2$	Exponential Terms <	t, sec. >	$\Delta\vartheta_1$, °C. <	$\Delta\vartheta_2$, °C. <	$\Delta\vartheta + \Delta\vartheta_2$, °C. <
0·2	0·315	$0{\cdot}05\theta_0$	1424	19·3	188·7	208·0
		$0{\cdot}02\theta_0$	1851	19·4	188·4	207·8
		0	∞	19·5	188·2	207·7
0·3	0·47	$0{\cdot}05\theta_0$	976	19·5	126·9	146·4
		$0{\cdot}02\theta_0$	1267	19·1	125·1	144·2
		0	∞	18·8	124·1	142·9
0·4	0·63	$0{\cdot}05\theta_0$	753	22·1	96·1	118·2
		$0{\cdot}02\theta_0$	976	21·4	93·9	115·3
		0	∞	20·6	92·6	113·2
0·5	0·79	$0{\cdot}05\theta_0$	619	23·9	77·5	101·4
		$0{\cdot}02\theta_0$	801	22·7	75·1	99·8
		0	∞	21·7	73·8	95·5
0·75	1·18	$0{\cdot}05\theta_0$	442	25·9	52·9	78·8
		$0{\cdot}02\theta_0$	569	24·0	50·6	74·6
		0	∞	22·3	49·4	71·7
1·0	1·57	$0{\cdot}05\theta_0$	353	29·8	40·2	70·0
		$0{\cdot}02\theta_0$	453	25·1	38·2	63·3
		0	∞	23·0	37·2	60·2
1·5	2·36	$0{\cdot}05\theta_0$	265	31·5	26·6	58·1
		$0{\cdot}02\theta_0$	338	27·9	25·3	53·2
		0	∞	25·1	24·7	49·8
2·0	3·15	$0{\cdot}05\theta_0$	221	34·0	19·7	53·7
		$0{\cdot}02\theta_0$	281	29·8	18·8	48·6
		0	∞	26·6	18·5	45·1

in a chamber of cross-sectional area $\sigma = 2{\cdot}2$ sq. ft. and height l. From the above it is known that conduction in the pellets is unimportant compared with the rate of heat transfer from gas to solid. The process can then be regarded as a counterflow heat-exchange process in which the pellets represent one fluid and the gas stream is the counterflowing fluid. Thus, to determine the mass flow of gas and optimum height of chamber for a given throughput of pellets, cross-sectional area of chamber, and temperatures of ingoing and outgoing pellets and gas, use can be made of the following relations[9]:

$$\theta_{10} = \theta_{11} - \frac{\left(\theta_{11} - \theta_{21}\right)\left[1 - \exp\left\{-A\alpha\left(\frac{1}{W_1C_1} - \frac{1}{W_2C_2}\right)\right\}\right]}{1 - \frac{W_1C_1}{W_2C_2}\exp\left\{-A\alpha\left(\frac{1}{W_1C_1} - \frac{1}{W_2C_2}\right)\right\}} \quad \ldots(10)$$

$$\theta_{20} = \theta_{11} - \frac{\left(\theta_{11} - \theta_{21}\right)\left(1 - \frac{W_1C_1}{W_2C_2}\right)}{1 - \frac{W_1C_1}{W_2C_2}\exp\left\{-A\alpha\left(\frac{1}{W_1C_1} - \frac{1}{W_2C_2}\right)\right\}} \quad \ldots(11)$$

$$W_1C_1\left(\theta_{11} - \theta_{10}\right) = W_2C_2\left(\theta_{20} - \theta_{21}\right) \quad \ldots(12)$$

where θ_{11} = gas inlet temperature (1450° C.)
θ_{10} = gas outlet temperature (200° C.)
θ_{21} = solid inlet temperature (0° C.)
θ_{20} = solid outlet temperature (1350° C.)
W_1 = mass flow of gas
W_2 = mass flow of pellets (1000 lb./hr.)
C_1 = specific heat (0·286 cal./g./° C. of burnt blast-furnace gas + 25% excess air at 800° C.)
C_2 = specific heat of pellet material (= 0·194 cal./g./° C.)
A = total surface area of pellets in chamber
α = heat-transfer coefficient between gas and pellets.

It will be noted that equations (10), (11), and (12) are not independent and that equation (12) can be obtained by suitable combination of the other two; *i.e.*, they are necessary and sufficient to determine two unknowns, which are in this case l, the height of the chamber, and W_1, the mass flow of gas. The remaining unknowns, A and α, can be represented in terms of l and W_1 as follows.

If the fractional voidage in the chamber is f (= 0·45, say) the total surface area of the pellets A is

$$A = \frac{3\sigma l(1 - f)}{a}$$

where the mean radius of the pellets is $a = 0{\cdot}635$ cm. The heat-transfer coefficient between gas and solid is taken as being equal to the heat conductance of the exchanger. Löf and Hawley[10] give the following empirical relation for this (in cal. per c.c. of bed per ° C. of temperature difference between gas and solid per second):

$$\alpha_1 = 1{\cdot}96 \times 10^{-2}\left(\frac{G}{2a}\right)^{0.7}$$

where G is the gas flow rate in g./sec./sq. cm. of bed cross-section. The heat-transfer coefficient or gas conductance is then given from this in cal./sq. cm./° C./sec. by

$$\alpha = 0{\cdot}65 \times 10^{-2} \frac{a}{1-f}\left(\frac{W_1}{2a\sigma}\right)^{0{\cdot}7}.$$

The value for the heat-transfer coefficient given by Saunders and Ford, which is probably more appropriate for the work, is given by

$$\alpha = 0{\cdot}56 \times 10^{-4} V,$$

where V is the velocity of the gas in cm./sec. measured at 17° C. and where the fractional voidage in the chamber is 0·45. Saunders and Ford worked with spherical particles and gas velocities of the order of 2 ft./sec., whereas the Löf and Hawley relation holds for particles of all shapes and somewhat lower velocities.

The above relation averages out the effect of particle size, but Löf and Hawley give the following expression for α_1 (in cal./c.c. of bed volume per ° C./sec.) obtained from the Saunders and Ford results which does take this effect into account:

$$\alpha_1 = 0{\cdot}152 \frac{G}{2a}.$$

From this α can be found as before in the form

$$\alpha = 2{\cdot}53 \times 10^{-2} \frac{W_1}{(1-f)\sigma} \text{ cal./sq. cm./sec./° C.}$$

This gives a much higher heat-transfer coefficient than does the Löf and Hawley expression.

Eliminating W_1 and substituting for A and α in equations (10)–(12) gives

$\alpha = 7{\cdot}137 \times 10^{-4}$ (Löf and Hawley), $ah = \frac{a\alpha}{K} = 0{\cdot}45$ or $\alpha = 2{\cdot}27 \times 10^{-2}$ (Saunders and Ford),

$$ah = \frac{a\alpha}{K} = 1{\cdot}44.$$

These values of ah come within the range discussed in the first section and the Saunders and Ford value, which is the more likely one, is well above the critical value discussed there. Inserting these values in equations (12) and (13) shows that the mass flow of gas is approximately 100 g./sec. The optimum height of chamber is about 2 ft., using Löf and Hawley's value for α, and 8 in. using the Saunders and Ford value, which is probably the more applicable.

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Aspects of Swedish Iron-Ore Concentration

By P. G. Kihlstedt

SYNOPSIS

The paper gives a survey of the present conditions influencing Swedish iron-ore beneficiation. The choice of iron contents in the concentrates obtained from different types of ore has been investigated in regard to the best possible adaptation for the coke blast-furnace process as applied in Sweden. To help in this study, an evaluation formula for concentrates is submitted, based on production costs of pig iron, and by this formula certain optimum calculations, comparisons between different types of ores, and 'cut-off' grade calculations have been carried out. The importance is discussed of high concentration for the utilization of Swedish apatite iron ores in domestic pig-iron production, which is mostly based on low-phosphorus raw materials, and for Swedish sponge-iron production. It is demonstrated that flotation methods would raise the iron recovery in the beneficiation of hematite ores. 912

Introduction

Concentration of Swedish iron ores does not only involve the purely technical problem of increasing the iron content of certain lean ores and parts of the ore bodies to raise them to the level of the richer ones, but has also developed into a link of both technical and economic importance between mining on one hand and extraction at the ironworks on the other. The ores available in the mines are treated in such a way that they become as suitable as possible for the subsequent metallurgical processes. Sweden has, however, many kinds of iron ores, and the development of these processes has caused continually varying demands to be made on the concentrated products. The most obvious requirement therefore of iron-ore beneficiation is that it should combine great adaptability with low operational costs.

Recently interest has been focused on the qualitative basis of the Swedish iron industry, a fact clearly reflected in several of the papers discussed in the 1940's at the annual technical conferences of Jernkontoret.[1–5] Since these discussions, expansion and development on new lines have taken place at Swedish ironworks, and production capacity of the mines has been greatly enlarged. These changes, together with a rapid development in dressing technique and

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sintering methods, have radically altered the conditions of Swedish iron-ore beneficiation, and the present paper is a further contribution to the discussion of this subject recently begun by Elfström.[6]

The present paper surveys the requisite conditions and possibilities of development of Swedish iron-ore concentration, and the simple economic calculations given are intended only as a provisional basis for further discussion, and as a help to those who want to apply similar calculations to their individual problems.

SWEDISH IRON-ORE RESOURCES AND PRINCIPAL FEATURES OF PRESENT CONCENTRATION METHODS

The character of the iron-ore resources is perhaps the most constant of the factors to be considered,

Table I

PROVEN IRON ORE RESOURCES OF SWEDEN*

a. Iron Ore Resources of Central Sweden (1943)[2]

All figures in metric tons × 10^6 O = Ore suitable for direct extraction C = Concentrate

Type of Ore		< 0·005% P	0·005–0·008% P	0·008–0·030% P	0·030–0·300% P	> 0·300% P	Total
I. Quartz iron ores (banded)	O	...	5·6	10·0	4·5	...	20·1
	C	...	1·6	6·4	0·9	...	8·9
							29·0
II. Quartz iron ores (not banded)	O	...	0·3	1·1	2·1	...	3·5
	C	...	13·1	0·7	10·1	...	23·9
							27·4
III. Skarn ores and calcareous ores < 1% Mn	O	...	2·4	2·6	0·2	...	5·2
	C	...	9·9	0·4	0·1	...	10·4
							15·6
IV. Skarn ores and calcareous ores > 1% Mn	O	5·6	4·7	3·4	...	...	13·7
	C	0·1	0·1	...	...	...	0·2
							13·9
V. Weathered ores > 1% Mn	O	...	...	7·4	...	...	7·4
	C	...	...	...	...	...	...
							7·4
VI. Pegmatite iron ores	O	...	...	...	...	...	...
	C	...	...	0·9	...	...	0·9
							0·9
VII. Apatite iron ores	O	...	...	...	...	110·7	110·7
	C	...	...	...	0·4	11·4	11·8
							122·5
VIII. Impregnated ores combined with apatite iron ores	O	...	...	...	2·6	...	2·6
	C	...	...	...	0·6	...	0·6
							3·2
Total	O	5·6	13·0	24·5	9·4	110·7	163·2
	C	0·1	24·7	8·4	12·1	11·4	56·7
O + C		5·7	37·7	32·9	21·5	122·1	219·9†

b. Iron Ore Resources of the Lapland Iron Ore Fields‡ (Kiruna, Tuolluvaara, Malmberget)

Apatite iron ores (1953)

Type	A	< 0·03% P	165
,,	B	0·03–0·1% P	110
,,	C	0·1–0·35% P	430
,,	C_2 + D	> 0·35% P	2285
		Total	2990

* As in Table II, figures for titaniferous iron ores and pyrite ores are excluded
† According to the latest estimate by the same authors[10] the resources in Central Sweden amount to 265,000,000 tons
‡ According to recent information from the mining companies

even if variations appear in connection with the exhaustion of certain ore bodies and the opening up of others. The quantities available in the two ore regions, Central Sweden and Lapland, are given in Table I. Table II is a general survey of the present iron-ore concentration, showing production in 1951 of ore directly suitable for smelting and of concentrate. Titaniferous iron ore and pyrite ores are not considered.

For 20 years Swedish pig-iron production has been based almost entirely on sintered concentrate, whereas the main part of the export ore is still desired in lump form. This is the reason for the typically Swedish practice of dressing the coarser sizes of the crushed ore for export grades and of concentrating the fines to higher grades principally for the home market. Magnetite ores are mainly sorted in magnetic separators, but the hematite ores require hand-picking or gravity separation. The operations are often combined with the production of macadam from the gangue.

Of the eight types of ore shown, the apatite iron ores dominate, forming about 97% of the total resources, exclusive of the titaniferous ores. The generally high phosphorus content predestines the apatite ores mainly for the production of commercial steel, and much is exported because of the large quantities available. At Kiruna the ore is still treated only by crushing, whereas at Malmberget and Grängesberg the ore is crushed and magnetically sorted or hand-picked. The finer grain sizes are at Grängesberg further dressed magnetically and by gravity separation, and from the fines certain types of concentrate are produced with either a very high or a specially low phosphorus content for the home market. At Malmberget the magnetic concentration of the fines is done in connection with grinding in several steps to obtain grades low in phosphorus, partly intended for the home market. At Blötberget the ore is concentrated magnetically and by gravity separation to fine-grained concentrates for the Domnarfvet Iron and Steel Works.

The next largest groups, the quartz iron ores (I and II in the table), together form about 2% of the resources. The greater part is exported, mainly because the ores are too siliceous for Swedish methods of extraction, and also are not suitable for high concentration because of their hematite content and fine-grained texture. Their low phosphorus content, however, makes them an important reserve for future production of high-grade steel.

At Stripa and Striberg lump ore down to about 6 mm. is treated by heavy media separation to raise the iron content to 50% for export, and the fines are concentrated in jigs and on tables to higher iron contents for the Swedish works. At certain export mines, however, such as Håksberg, the entire ore is obtained as fine-grained products as the ore in coarser sizes cannot be sufficiently concentrated.

The skarn and calcareous iron ores are the real home-market ores with low phosphorus contents and with the iron generally in the form of magnetite. These ores are generally suitable for high concentration, but in this process the manganese and lime

Table II

IRON ORE PRODUCTION IN SWEDEN, 1951

All figures of weight in metric tons × 10^3 O = Ore suitable for direct extraction C = Concentrate

Type of Ore		< 0·005% P			0·005–0·008% P			0·008–0·012% P			0·012–0·030% P			0·030–0·3% P			> 0·3% P			Total Wt.
		Wt.	Fe, %	P, %	Wt.	Fe, %	P, %	Wt.	Fe, %	P, %	Wt.	Fe, %	P, %	Wt.	Fe, %	P, %	Wt.	Fe, %	P, %	
I. Quartz iron ores (banded)	O							155	49	0·008	274	46	0·021							429
	C				6	66	0·007	114	64	0·010	22	54	0·014							142
																				571
II. Quartz iron ores (not banded)	O				9	65	0·006				68	46	0·016							77
	C	49	66	0·004	6	64	0·005	11	64	0·010				71	65	0·032				137
																				214
III. Skarn ores and calcareous ores < 1% Mn	O				97	50	0·005				79	53	0·020							176
	C	72	61	0·004	188	64	0·005	55	65	0·009										315
																				491
IV. Skarn ores and calcareous ores > 1% Mn	O	127	46	0·004				100	48	0·009	69	38	0·018							296
	C				12	65	0·005				1	63	0·018							13
																				309
VI. Pegmatite iron ores	O													7	38	0·100				7
	C										53	61	0·017							53
																				60
VII. Apatite iron ores	O										407	67	0·021	1924	65	0·138	10,618	60	1·315	12,949
	C							35	72	0·009	175	71	0·025	113	65	0·189	8	34	6·430	331
																				13,280
Total	O	127	46	0·004	106	51	0·005	255	49	0·008	897	55	0·020	1931	65	0·138	10,618	60	1·315	13,934
	C	121	63	0·004	212	64	0·005	215	66	0·009	251	67	0·022	184	65	0·128	8	34	6·430	991
Total O + C		248	54	0·004	318	60	0·005	470	56	0·008	1148	58	0·020	2115	65	0·137	10,626	60	1·315	14,925

contents are lost, and concentration is therefore often avoided. Market conditions often cause the manganiferous iron ores to be exported. They are treated almost wholly by crushing combined with coarse magnetic sorting, but otherwise such sorting of the ores for the home market is done only as a step in the graded crushing and concentrating to products suitable for extraction. Magnetic concentration dominates as the ore mineral is generally magnetite; in certain cases the process is combined with flotation of sulphides. The end product is, of course, a fine-grained concentrate.

The aims of Swedish concentration may be summarized as follows:

(i) To turn the ore into a form suitable for sintering or for direct smelting
(ii) To remove the content of gangue, partially or as completely as possible
(iii) To remove or concentrate the phosphorus content of the ore in connection with the iron concentration
(iv) To remove, and in certain cases recover, the sulphide content of the ore.

In the following, only production of concentrates for the home market will be discussed. The first object will be to find a method of appraisement of the ore treatment, comprising an evaluation of the composition of different concentrates from the point of view of the ironworks.

EXTRACTION PROCESSES AND OPTIMUM SLAG CONTENTS

The only way to adjust the concentrating processes correctly and to obtain a reliable evaluation of different grades is to make overall economic calculations and to weigh gains and costs against each other.

In the non-ferrous field the economic relation between mining and smelting has traditionally been established by means of formulæ where the costs and yields of the smelting plant are taken into account together with metal prices. By means of these formulæ the most economical grade of concentration can be calculated and the process can be regulated accordingly. The formulæ show the economic value to the smelting works of increased concentration and also the importance of the removal of various impurities.

Selling formulæ, based on the market price for pig iron, were formerly used for iron ores also. The reason why such formulæ are no longer used is probably that the price of pig iron is low compared with that of non-ferrous metals and is therefore much more influenced by local conditions such as freights and slag formation.

Two years ago C. G. Cedervall of the Jernkontoret Research Organization and the author started to investigate the question of evaluation of iron-ore concentrates at a constant pig-iron price, hoping to indicate the best application of the concentration processes, and attempted, avoiding commercial considerations, to elucidate the connection between the treatment of the ore at the mines and concentration plants and the further treatment of the concentrates at the iron works.

Evaluations were made for a coke blast-furnace, as coke pig iron represents about 70% of the iron produced from ore in Sweden today. Conditions for production of electric pig iron and charcoal pig iron have not been treated in detail. Altogether nine calculations of ore burdens were made for different types of concentrate. Not only the iron content but also the contents in the concentrate of SiO_2, CaO, and MgO, as well as the basicity $(CaO + MgO)/SiO_2$, were taken into account. The nine types of concentrate burdens had iron contents of 55%, 60%, and 65% respectively, and the basicities were those of a self-fluxing concentrate corresponding to about 1·50, 0·78, and 0·25. A normal value for basicity of the slag was fixed at 1·31, and that of the coke ash was assumed to be 0·18 at an ash content of 10%.

The basicity of the coke blast-furnace slag must be high as it should take up the sulphur content of the coke, and for the same reason the slag quantity should be as high as about 450 kg. per ton* of pig iron so as not to obtain too high a sulphur content in the slag. Thus almost all Swedish iron ores have a gangue basicity lower than that corresponding to the self-fluxing basicity of about 1·50 and so require an addition of lime to the mixture. The siliceous concentrates from the quartz iron ores with a basicity of about 0·25 do, of course, require specially high additions of lime.

The calculation of ore burden was made for each concentrate independently and therefore gave higher additions with more siliceous concentrates. This can be seen in Fig. 1, where the slag quantities per ton of pig iron found by calculations of ore burdens are plotted against concentrate content and gangue basicity.

The set of curves shown in Fig. 2 were obtained in such a way that the iron contents from Fig. 1 were determined, at different gangue basicities, for slag quantities of 200, 300, 350, and 450 kg./ton of pig iron. Each curve thus represents iron contents at equal slag volume. At basicities above 1·50 a certain excess quantity of CaO + MgO is assumed to remain in the concentrate.

The lower the slag quantity per ton of pig iron that can be allowed, the higher is the iron content of the concentrate. At a basicity of 1·50, 450 kg. of slag corresponds to an iron content in the concentrate of 54·2%, and 350 kg. of slag to an iron content of 57·7%. At a gangue basicity of 0·25 the iron contents are 61·3 and 63·4%, respectively. If the optimum slag quantity is taken to be normally 450 kg./ton of pig iron for coke blast-furnaces, and if 350 kg. represents an extreme limit, the iron contents of the concentrates for the basicities mentioned should vary between 52 and 61%, according to the curve for 450 kg. of slag.

The iron contents of this curve indicate a suitable concentration at the basicities in question. The values mentioned hardly inspire an interest in high concentration of the ores.

The preceding shows that the sulphur absorption of the slag to a certain degree directs the aim of the concentration. If desulphurization of the coke pig iron can be done after the blast-furnace process, as in Kalling's method in a rotary furnace,[7, 8] the case

* Ton in this paper is always metric ton.

is fundamentally changed. The slag quantity may then be greatly reduced, normally to about 300 kg./ton of pig iron, with 200 kg. as an extreme limit. As only about half this slag content comes from the ore and the rest comes from the coke ash, there is a far wider margin for removing gangue material by means of concentration, a fact which ought to be of economic importance. As at the same time the most suitable basicity of the slag will be lower, certain savings of lime may be obtained, even if the self-fluxing mixture, because of changed proportions, still has an approximate basicity of 1·50.

Figure 2 shows that the iron contents of concentrates at 300 kg. of slag per ton of pig iron vary from 59·5% at a basicity of 1·50 to 64·5% at a basicity of 0·25. This means improved concentration conditions.

The ore burdens of the blast-furnaces, however, generally consist of mixtures of several concentrates, and it is possible to vary the degree of concentration of the ores. The treatment of each individual ore is thus not fixed by Fig. 2, but many other considerations may influence the determination of the iron contents of the different concentrates, *i.e.*, the decision on how much gangue should be left in the concentrate at grinding and concentration. Such considerations may be of the following types:

(i) From which ores may silica be removed at the lowest concentration cost ?

(ii) Which ores give the highest iron yield in high concentration ?

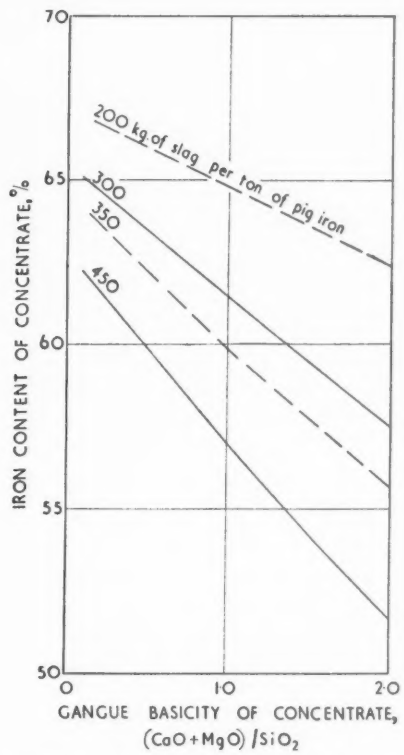


Fig. 2—Iron contents at equal slag volume for concentrates with different gangue basicities

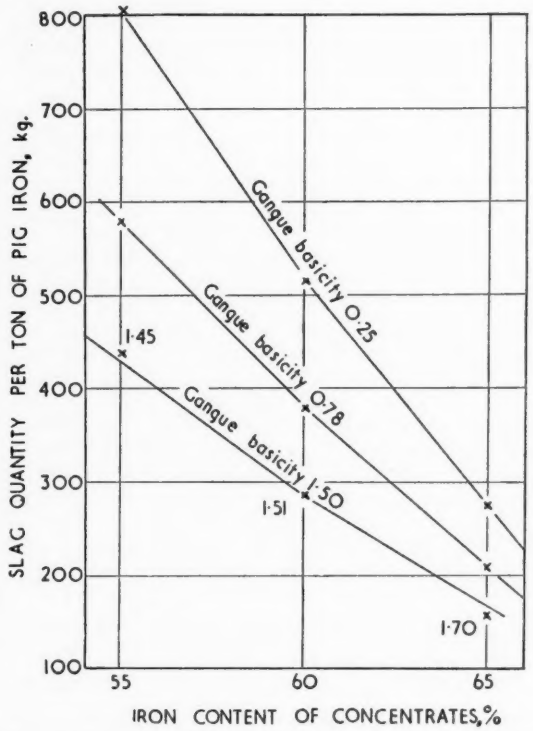


Fig. 1—Relation between specific slag quantity and iron content of concentrate

(iii) Which concentrates have the heaviest transport costs ?

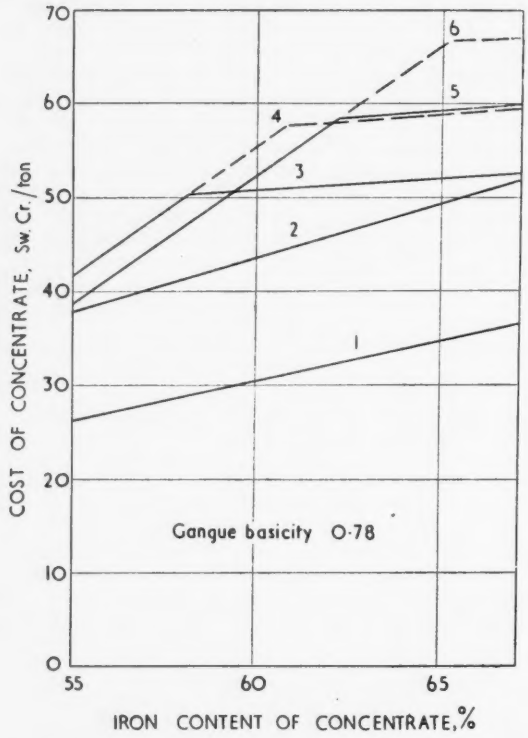
(iv) Which ores are most suitable for high concentration with regard to removal of other impurities, such as phosphorus and sulphides ?

(v) Which ores have the most favourable composition for slag formation ?

EVALUATION OF IRON-ORE CONCENTRATES WITH REGARD TO COST OF SLAG FORMATION IN THE BLAST-FURNACE PROCESSES

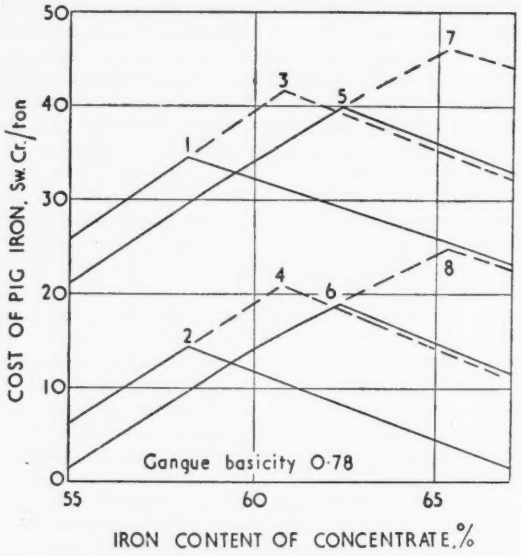
The fundamental formula for evaluation of concentrates aims at calculating the various costs which concentrates with different compositions cause in pig-iron production. We have thus started from a fixed pig-iron price, deducted the costs of smelting, and calculated the balance to pay for the different concentrates. We have then analysed the cost functions with regard to the compositions of the concentrates and obtained a formula.

Certain simplifications have been necessary. The coke consumption has been assumed to increase by 40 kg. at an increase of 100 kg. in the slag amount, a value which is correct, according to the literature, for different foreign coke blast-furnaces at higher slag contents, and which has also been considered to be applicable to Swedish conditions. However, the Domnarfvet Works have pointed out that in their experience the gain in coke consumption by reduced



Costs of concentrate at (1) 40%, and (2) 30% Fe in raw ore
Value of concentrate in normal blast-furnace process: (3) 450 kg., and (4) 350 kg. of slag per ton of iron
Value of concentrate in blast-furnace process with subsequent desulphurization: (5) 300 kg., and (6) 200 kg. of slag per ton of iron

Fig. 3—Values and costs of concentrates of magnetite ore with different iron contents



Optima for ordinary blast-furnace process:
(1) and (2) 450 kg. of slag per ton of pig iron with (1) 40% and (2) 30% Fe in raw ore
(3) and (4) 350 kg. of slag per ton of pig iron with (3) 40% and (4) 30% Fe in raw ore
Optima for blast-furnace process with subsequent desulphurization:
(5) and (6) 300 kg. of slag per ton of pig iron with (5) 40% and (6) 30% Fe in raw ore
(7) and (8) 200 kg. of slag per ton of pig iron with (7) 40% and (8) 30% Fe in raw ore

Fig. 4—Optimum curves for concentration of magnetite ore to different iron contents. Total economic margin for production process from ore in mine to pig iron

slag quantity is not quite so high. As the slag basicity was fixed at 1·31 the increase in coke consumption at increased content of gangue in the concentrate is directly proportional to the SiO_2 content in the concentrate, which means that the increase was 10·2 kg. of coke for each 1% of SiO_2 and ton of concentrate. In the same way they found that 'other smelting costs' increase proportionally to the SiO_2 content of the concentrate.

If the costs of loading and transport of the concentrate from the mine are included in the formula, the following general formula for the normalized concentrate value at the mine is:

$$V_{conc.} = U \times Fe + 18\,K\,(CaO + MgO) - (26{\cdot}8\,K + 10{\cdot}2\,B + 12{\cdot}75\,D)SiO_2 - T\frac{100}{100 - H_2O}$$

where $V_{conc.}$ = Normalized concentrate value, Swedish Crowns per ton dry weight
U = Unit value for iron in theoretically gangue-free concentrate
Fe = Iron content, %
K = Lime price, Sw.Cr. per kg.
$CaO + MgO$ = Combined CaO and MgO contents, %
B = Coke price, Sw.Cr. per kg.
D = Other smelting costs, Sw.Cr. per kg. of pig iron
SiO_2 = Silica content, %
T = Loading and freight costs, Sw.Cr. per ton of concentrate
H_2O = Moisture content, % of wet weight.

For the following calculations it is assumed that:

Value of concentrate at iron plant for self-fluxing 60% Fe concentrate	= Sw.Cr. 70 per ton dry weight
Price of limestone	= Sw.Cr. 20 per ton
Price of coke	= Sw.Cr. 137 per ton
Loading and freight costs	= Sw.Cr. 8 per ton
Moisture of concentrate	= 5% H_2O.

The formula is then:

$$V_{conc.} = 1{\cdot}35\,Fe + 0{\cdot}36\,(CaO + MgO) - 2{\cdot}44\,SiO_2 - 8{\cdot}40.$$

For the ensuing calculations it is further assumed:

Cost of Kalling's desulphurizing process	= Sw.Cr. 5 per ton of pig iron

This gives the formula:

$$V_{conc.}(K) = 1{\cdot}30\,Fe + 0{\cdot}36\,(CaO + MgO) - 2{\cdot}44\,SiO_2 - 8{\cdot}40.$$

A possible saving of limestone has not been taken into account. At too low a slag quantity the ore mixture may be corrected by adding a self-fluxing material at a price of Sw.Cr. 20 per ton. It has been assumed that the concentrate is produced by magnetic concentration as follows:

Operation	Cost, Sw.Cr./ton of raw ore
Mining, crushing, sorting, etc.	15
Concentration to 55% Fe	3
Concentration to 65% Fe	5

For other contents, proportional concentrating costs are obtained. The iron contents in tailing at 55% and 65% in concentrate are, respectively, 7% and 6%.

ECONOMIC CALCULATIONS OF OPTIMUM IRON CONTENTS IN CONCENTRATE WITH REGARD TO SLAG-FORMING COMPONENTS IN COKE PIG-IRON SMELTING

The first economic calculation using the above assumptions is an optimum calculation for charging with concentrate of basicity 0·78 and with different iron contents. In Fig. 3 the evaluations found by the formula have been plotted for different iron contents in the concentrate at a constant pig-iron price, adjusted for the additional cost of extra slag-forming material. The diagram contains two curves, one for ordinary coke pig iron and one for desulphurized pig iron, the former being drawn both for the normal slag quantity of 450 kg. and for the extreme low quantity of 350 kg./ton of pig iron. Similarly, the latter curve has been drawn for the slag quantity of 300 kg., and also for the perhaps unobtainable extreme low quantity of 200 kg. per ton of pig iron. Against these concentrate values at constant pig-iron price have been plotted the production costs of the concentrates according to the assumptions made. This has been done for two ores, one containing 30% and the other 40% of iron. The difference between the value curves and the cost curves shows graphically the economic margin per ton of concentrate for the whole production process from ore in the mine to ready desulphurized pig iron.

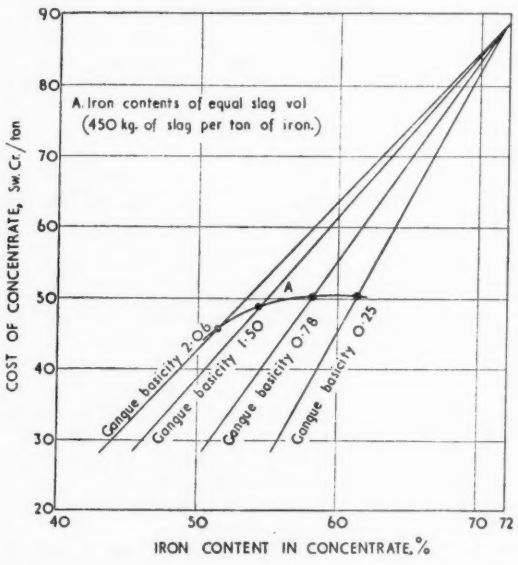


Fig. 5—Evaluation curves at constant pig-iron cost for magnetite concentrates with different basicities and iron contents, according to formula for ordinary coke blast-furnace process

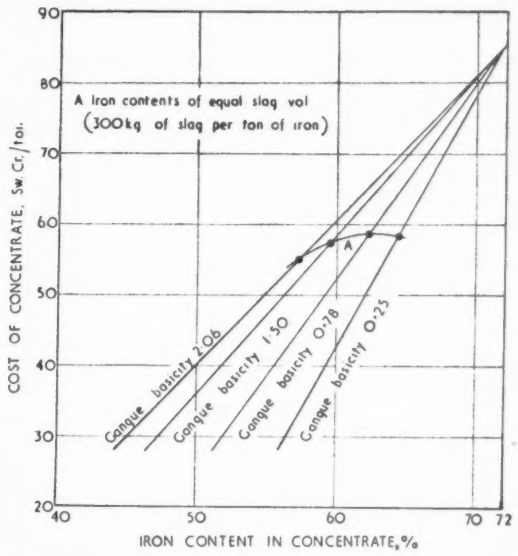


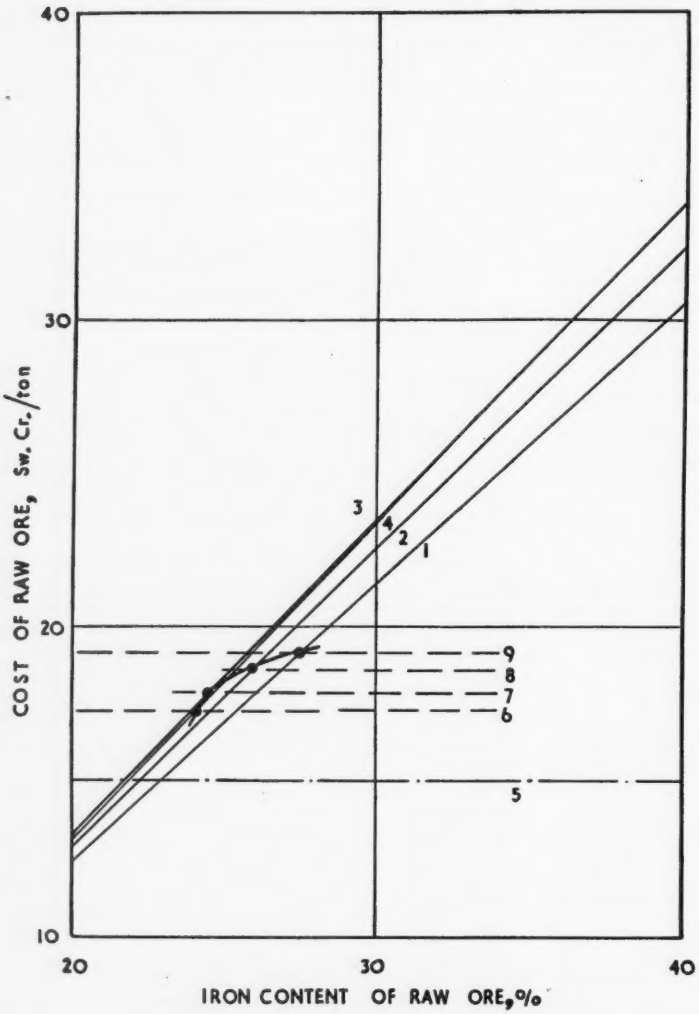
Fig. 6—Evaluation curves at constant pig-iron cost for magnetite concentrates with different basicities and iron contents, according to formula for blast-furnace process with subsequent desulphurization

The curves in Fig. 4, where these margins have been calculated in Sw.Cr. per ton of pig iron produced, show that it is of great economic importance for the ore mixture to be chosen in such a way that the optimum slag quantity is obtained. The optimum economical result using the desulphurization process is seen to be more profitable than using the ordinary coke pig-iron process by Sw.Cr. 5 per ton of pig iron. With highly concentrated ores, a better economic result (as much as Sw.Cr. 10 per ton of pig iron) would be obtained if the desulphurizing process were used with a slag quantity of only 300 kg./ton than if the ordinary pig-iron process were employed and slag-forming additions lowered the iron content to an extent corresponding to a slag quantity of 450 kg./ton of pig iron. This calculation may be important in the future when larger quantities of the highly

concentrated low-phosphorus A–10 Grängesberg product will be available.

The evaluation formulæ for concentrate at constant pig-iron cost give far higher values for the calcareous concentrates than for the siliceous, as is seen from Figs. 5 and 6, where formula values at different iron contents are shown for four different types of concentrate with the following gangue contents at 60% Fe:

	CaO, %	MgO, %	SiO_2, %	Basicity
(1)	1·72	1·14	11·46	0·25
(2)	4·71	1·57	8·04	0·78
(3)	7·15	1·49	5·72	1·50
(4)	9·40	1·50	5·30	2·06



(1–4) Values of concentrate per ton of raw ore according to formula, with gangue basicity (1) 0·25, (2) 0·78, (3) 1·50, and (4) 2·06
(5) Cost exclusive of concentration
(6–9) Total cost per ton of raw ore when concentrating to an iron content of (6) 51·4%, (7) 54·3%, (8) 58·2%, and (9) 61·3%

Fig. 7—Values and costs of concentrates of magnetite ores after concentration to iron contents of equal slag volume, using ordinary coke blast-furnace process, with 450 kg. of slag per ton of pig iron

Figures 5 and 6 show that the concentrates containing 55% Fe are evaluated as follows according to the formulæ used:

		Cost, Sw.Cr./ton of concentrate Coke Blast-Furnace Process	
	Basicity	Ordinary Process	With Desulphurization
(1)	0·25	27·60	24·90
(2)	0·78	41·30	38·50
(3)	1·50	50·40	47·50
(4)	2·06	53·00	50·40

The siliceous concentrate has thus only half the value of the calcareous one if the calculation is based on a constant value of the pig iron. This difference in value, however, is compensated if the smelting conditions are adjusted to the optimum slag quantities, and if the siliceous concentrates are more highly concentrated to obtain the same specific slag quantity. In Figs. 5 and 6 the optimum iron contents in the concentrate for 450 kg. of slag per ton of pig iron at the ordinary coke blast-furnace process, and 300 kg. of slag per ton of pig iron at the desulphurizing process according to Kalling, have been plotted; the values of the different types of concentrates are seen to be equalized in this way.

Provided that the different concentrates are treated so as to obtain the respective optimum contents, and are given the corresponding values, the economic conditions may be graphically read by plotting gains and costs as in Figs. 7 and 8 where all values and costs are calculated per ton of raw ore.

This type of diagram is of great interest as it explains the economic conditions of mining. On one hand there are the costs of the ore treatment, which are, on the whole, the same for all iron contents of the ores, up to the treatment of the concentrate; the costs may, to a certain extent, be proportional to the ore volumes and thus are somewhat higher per ton for a lean ore than for a rich one, but this has been disregarded in these calculations. On the other hand the income, *i.e.*, the value of the concentrates produced, is on the whole proportional to the iron contents of the ores. The economic margin will, therefore, increase with the increase in iron content of the treated ore.

If the curves are followed towards lower iron content in the treated ore, the intersection point between income and expenditure is reached. At the iron content

in the ore corresponding to this intersection point the total expenditures are just covered by the value of the concentrate produced. At a somewhat lower iron content a new intersection point may be constructed between the sum of the variable costs and the value of the concentrate. Below this iron content, mining is carried out at a direct loss. There is, however, reason to let the first intersection point represent the limit of analysis. This so-called 'cut-off' grade is, of course, of fundamental importance for every mining enterprise.

The economic margin which can be obtained graphically from Figs. 7 and 8 for different iron contents in the ore, different basicities of the concentrate, and the two pig-iron processes, is shown in Fig. 9. Two groups of curves have been drawn, *viz.*, for production of concentrate from raw ore with 30 and 40% Fe, respectively. The calcareous ore is found to have an economic margin of Sw.Cr. 2·50–5 per ton of raw ore larger than the siliceous. The blast-furnace process followed by desulphurizing by the Kalling process has a margin of Sw.Cr. 1–2 per ton of raw ore larger, the difference being greater for siliceous than for calcareous ores.

Figure 10 shows the 'cut-off' grades found from Figs. 7 and 9 for different basicities of the concentrate gangue and for the two pig-iron processes. At iron contents of equal slag volume in the concentrates produced, 'cut-off' grades of about 25% Fe are obtained for the magnetite ores investigated. They are a few per cent. higher for the siliceous ores than for the calcareous, and ½–1% lower using Kalling's desulphurizing method than by the ordinary pig-iron process.

The following general conclusions in regard to treatment of home-market concentrates intended for extraction may be drawn, provided that no special freight or ore conditions invalidate the calculations:

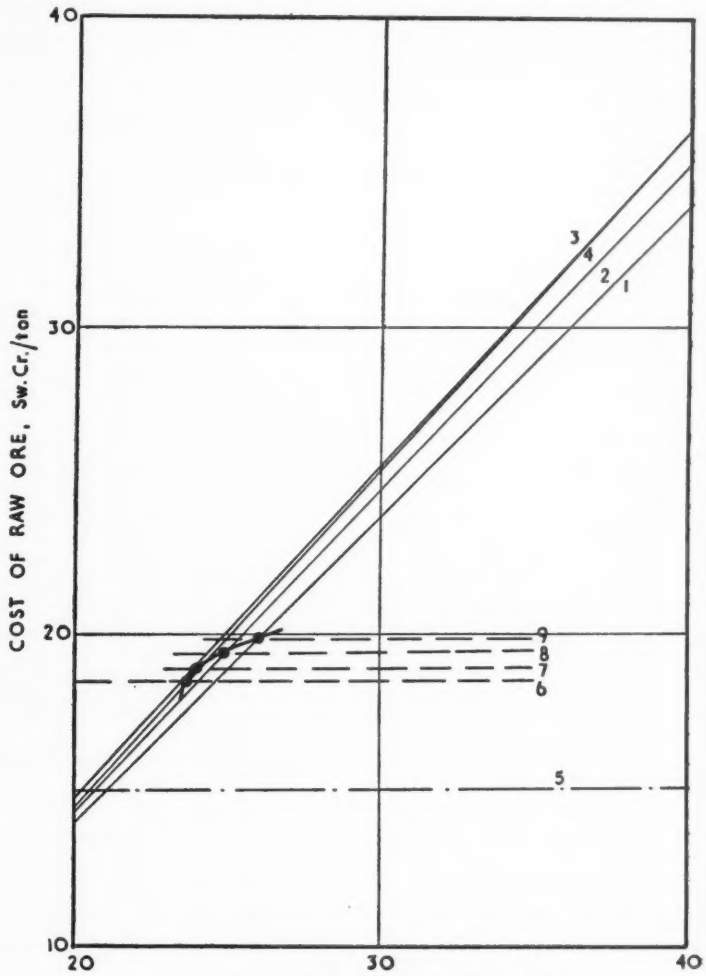
(1) It is economically important that the average iron content of the burden used in extraction corresponds to the suitable slag quantity. There is also a marked optimum iron content for the burden.

(2) For each concentrate there exists an iron content of equal slag volume which may, to a certain extent, serve as a point of aim in choosing the grade of concentration. The different concentrates should thus be more highly concentrated the more siliceous they are. In this way their lower value is compensated.

(3) By lowering the slag content and by desulphurizing after the blast-furnace process the economic conditions for high concentration are improved. This is especially true for siliceous ores.

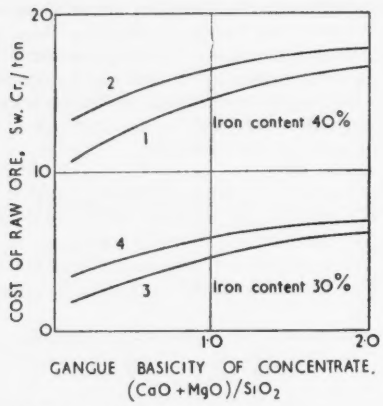
INFLUENCE OF OTHER CONCENTRATE COMPONENTS ON CHOICE OF IRON CONTENT

It is, however, not only the slag-forming components which influence choice of iron content of the concentrate. The value of the concentrates is judged also



(1–4) Values of concentrate per ton of raw ore according to formula, with gangue basicity (1) 0·25, (2) 0·78, (3) 1·50, and (4) 2·06
(5) Cost exclusive of concentration
(6–9) Total cost per ton of raw ore when concentrating to an iron content of (6) 57·3%, (7) 59·5%, (8) 62·3%, and (9) 64·5%

Fig. 8—Values and costs of concentrates of magnetite ores after concentration to iron contents of equal slag volume; using blast-furnace process with subsequent desulphurization, with 300 kg. of slag per ton of pig iron



(1) and (3) Ordinary coke blast-furnace process
(2) and (4) Blast-furnace process with subsequent desulphurization

Fig. 9—Total economic margins for coke pig-iron production at different basicities and iron contents in raw ore

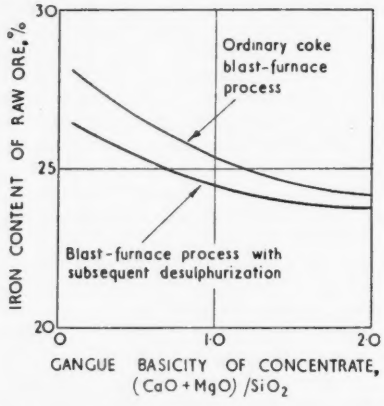


Fig. 10—'Cut-off' grades for magnetite ores of different basicities

from the content of certain components influencing quality of the product, such as manganese, phosphorus, copper, and sulphur.

The manganese content of the manganiferous iron ores is bound as silicate or carbonate minerals to the gangue and is therefore more or less lost in concentration. As a rule the manganiferous iron ores proper are therefore not concentrated in any other way than by magnetic sorting, by which waste rock is removed in coarse sizes. The more siliceous ores are generally exported; the calcareous and the almost phosphorus-free ores, such as Dannemora, Klackberg, and Kolningberg, have long been of great value for the production of high-grade steel. With iron contents of only 44–48% they are of special importance for slag composition.

Only about 13% of ingot steel production in 1951 was obtained by the basic Bessemer process. Even if production of basic Bessemer steel increases in the future the home market for the phosphorus-rich ores will not be large. Concentrates used for the production of quality steels are treated in such a way that a grade is obtained with a phosphorus content of about 0·010% or lower.

An interesting treatment, although carried out on a relatively small scale, is the concentration of phosphorus fines at Grängesberg, where, by the only flotation process for iron ore in Sweden, the iron and apatite contents of the fines are concentrated together to a product containing 34% of iron and 6·5% of phosphorus.[9] This product is used in the making of basic Bessemer pig iron.

Lowering the phosphorus content of the raw ore to obtain a concentrate with low phosphorus content is becoming more frequent. In magnetite concentration this method is generally used in conjunction with the removal of the gangue, and with higher phosphorus contents in the raw ore high concentration may be required. In gravity separation the dephosphorizing is not as good, since the specific gravity of the apatite is as high as about 3·2, and this mineral is, therefore, to a certain extent concentrated with the iron ore. In flotation of iron-ore minerals the apatite may be influenced in either an activating or depressing direction, but in the simplest flotation process the phosphorus content is concentrated with the iron content. Mention should, however, be made of Bring's comprehensive work on phosphorus depression in hematite flotation.[10–13]

How much the phosphorus content in a magnetite concentrate may be lowered depends on the phosphorus content of the magnetite mineral itself, and on the possibilities of complete liberation of the magnetite and apatite grains by grinding, in so far as the phosphorus content of the gangue is assumed to be bound entirely to this mineral.

Figure 11 shows the lowering of the phosphorus content in connection with concentration of the iron content of some of the Central Swedish iron ores poor in phosphorus when concentrated magnetically. The analyses are taken from the official Swedish statistics. The iron mineral in some of the ores is apparently not quite free from phosphorus, but the tendency to dephosphorize is evident. As the P/Fe ratio decreases

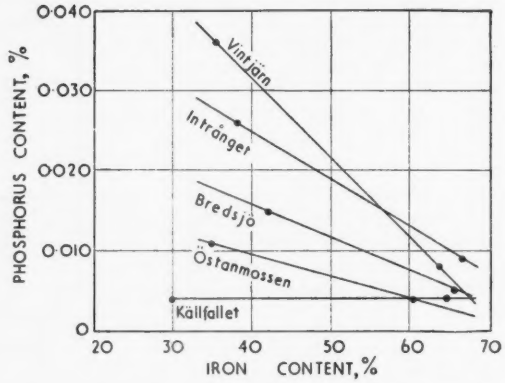


Fig. 11—Relation between phosphorus and iron content in magnetic concentration of some Central Swedish magnetite ores

even faster than the absolute value of phosphorus with the grade of concentration, it is clear that high concentration would in this case be useful.

This is still more evident for the apatite-rich ores. To transform some portion of these ores to a concentrate suitable for the main part of the Swedish iron and steel industry is one of the big tasks of Swedish beneficiation technique.[14] This problem has been solved in an excellent way by the Grängesberg concern, both at Malmberget and at Grängesberg, and also by the Tuolluvaara Gruv AB. When the production of A–10 concentrate at Malmberget and Grängesberg reaches full capacity, and when the new concentration plant at Tuolluvaara is completed, the iron industry will have very good raw materials available in large quantities. However, the long transports in connection with the demand for low phosphorus content have made such highly concentrated products necessary that the main problem will probably be to find suitable additions for slag formation and also to adapt extraction processes to the new conditions. Figure 12 shows the relation between increase in iron content and decrease in phosphorus content caused by magnetic concentration of Malmberget and Tuolluvaara ores as well as by concentration and dephosphorizing by leaching with nitric acid at Grängesberg. Lowering the phosphorus content leads to a very high iron content, corresponding to concentrates almost entirely gangue-free.

At Malmberget and Tuolluvaara this is done, or will be done, in connection with a fairly fine grinding of the magnetite concentrate, and the process is possible because the magnetite grains of these ores are fairly easily liberated. In Grängesberg the concentrate is fairly coarse, as dephosphorizing can be done on an ore highly concentrated in a coarser stage.

If the iron ore has a high sulphide content, it is easy to remove the sulphide more or less completely by xanthate flotation. This, however, requires grinding to liberation and flotation grain size and, by a fairly complete magnetite separation, a high concentration of the magnetite concentrate is obtained at the same time. The best example of this method is at Bodås where, in addition to the 69% magnetite concentrate, a good pyrite concentrate is also obtained. But in Central Sweden there are also iron ores containing copper, zinc, and lead sulphides. Best known is the Bäckegruvan ore containing 1% of copper, which is floated and magnetically concentrated to a content in the magnetite concentrate of 66% Fe.

The separation of apatite and sulphides from Swedish iron ores may thus constitute a stronger inducement to high concentration than would really suit the coke blast-furnace process. Whereas other countries generally have low-grade iron ores, which are difficult to concentrate and are therefore mixed with rich ore to lower the specific slag quantity, in Sweden rich raw materials will be available to such an extent that the ironworks would have to mix them with suitable local ores to obtain the necessary slag quantity.

This happens because Sweden has a large supply of easily concentrated magnetite ores. In this situation the less easily concentrated, but almost phosphorus-free, hematite ores will also have a chance on the home market as they may not need to be concentrated to such high iron contents.

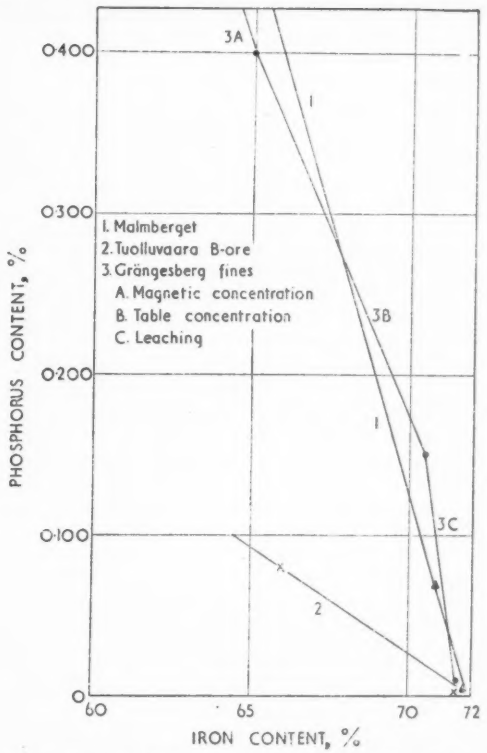


Fig. 12—Relation between phosphorus and iron content in magnetic high concentration of Malmberget and Tuolluvaara ores and in dephosphorizing of Grängesberg fines

GRAIN-SIZE DISTRIBUTION OF CONCENTRATES AND INFLUENCE OF SINTERING METHODS ON CHOICE OF IRON CONTENT

In the preceding the sintering properties of the concentrates and their influence on the choice of iron content in the concentrate have not been considered. Until recently this influence has, however, been wide and has had a restraining influence on high concentration. It has been shown that at constant vacuum there is a relationship between sintering time in pan-sintering and content of grain sizes — 200 mesh.[15] As an increased iron content of the concentrate generally means increased liberation obtained by finer grinding, a higher iron content would mean increased sintering costs. It is not easy to state how large the increase is, but this fact has in many cases greatly influenced concentration processes.

The introduction of pelletizing has changed the situation for the most finely ground concentrates. Research work has brought a precise knowledge of requisite conditions for pelletizing in regard to the grain-size distribution and specific surface of the concentrate.[16, 17]

Modern concentration technique could influence the working conditions of pan-sintering in many ways. Thus by rod-mill grinding, as well as by grinding with

small balls in closed circuit with classifier, much more favourable grain-size distributions can be obtained than formerly. The ore minerals may in this way be crushed to liberation with considerably smaller slime formation. The changed grain-size distribution is not only reflected in the weight-percentage of — 200 mesh in the screen analysis, but this should be combined with measurement of the specific surface.

The concentrates can also be washed in hydrocyclones, being thus divided into a larger quantity for pan-sintering and a smaller quantity for pelletizing. For pan-sintering the removal of the fine-grained material reduces the air resistance and thereby increases the sintering capacity, whereas pelletizing treats the fine material with high specific surface.

Sintering of fine-grained rich-hematite concentrates is a problem which probably requires detailed investigation before the flotation processes can be applied with full advantage. Would the fine-grained hematite concentrates be suitable for pelletizing, and if so, could they be sintered only if mixed with larger quantities of magnetite concentrates?

Several pelletizing plants are now being built, but the main part of the sintering capacity in Sweden will continue to consist of pan-sintering plants, with the inherent difficulty of receiving fine-grained concentrates. Improved grinding technique, especially rod-mill grinding, may be important in connection with the further development of high concentration.

HIGH CONCENTRATION OF MAGNETITE FOR PRODUCTION OF SPONGE IRON

In pig-iron manufacture the iron-ore concentration involves difficult adjustment problems. The optimum iron contents are generally much lower than those technically obtainable, and a complete liberation of the ore minerals is often not desired. Otherwise, the tendency in beneficiation is to obtain the valuable minerals in as pure a condition as possible with all the qualitative advantages this brings in the subsequent metallurgical processes, since a purification of the metal by beneficiation is normally cheaper than by metallurgical processes.[14]

There is another part of iron production where the purification of the ore by concentration methods has unlimited possibilities of development, *viz.*, sponge-iron production. As the gangue left in the sponge iron must be converted to slag in the O.H. or electric furnace, where costs of slag formation are considerably higher than in the blast-furnace, the highest possible purity of the sponge iron is required. So far only magnetite concentrate, obtainable in an almost entirely clean state, has been used. As sponge-iron production in Wiberg's method is combined with pelletizing and in the Höganäs method is based on fine-grained concentrates, fine grinding causes no difficulties.

Laboratory experiments have shown that the separation for a very high grade of concentration would be best in an alternating field with drum separator and with the material dry, a method which may be able to compete with graded wet magnetic separation, as the alternating field causes a constant rearrangement of the grains and as the centrifugal force of the drum contributes to the ejection of the gangue and mixed grains. The main difficulty is that the dry material causes dust problems.

It is not yet absolutely certain that in sponge-iron production and pelletizing complete freedom from gangue minerals in the concentrate is the most favourable condition. In regard to the subsequent steel production in basic furnaces, the only gangue component that is absolutely harmless is lime. From this point of view calcareous ores may perhaps be preferred as raw material, and it would help to mix burnt lime with the concentrate to improve pelletizing. The techniques of these new processes, not yet completely stabilized, show many possibilities of variation.

IRON RECOVERY AT CONCENTRATION OF SWEDISH ORES

The problems in connection with metal recovery in beneficiation processes may be of two kinds, those capable of economic calculation, where the maximum gain obtainable at the moment is taken as a standard, and the less definite views on the future, in which the best way of utilizing the natural wealth of the country should be considered in relation to the fact that the deposits will only be available once. The former type needs no comment; the latter, however, contains several factors which may influence beneficiation methods.

In regard to the low recoveries obtained on concentrating iron ores containing iron silicates in the gangue, if the gangue mineral of an ore contains 5–10% iron as silicate, this should not influence the process, as is often thought, except that the gangue remaining after concentration needs to have a somewhat higher value for pig-iron production. Otherwise, only the part of the iron content that could be concentrated should be taken into consideration. Rocks with 5–10% iron are present everywhere in nature and have not, and are in Sweden never going to have, any special value. A beneficiation process with a lower yield as a result of the removal of iron bound as silicate to a moderate extent should not for this reason be ignored.

With hematite concentration the case may be different, as this dressing has always been done by gravity methods which have never given good yields in the finer grain sizes. As the hematite ores are often siliceous and especially fine-grained, their concentration to iron contents acceptable to the Swedish market is made very difficult by lower yields. The iron contents of the tailing lie between 12 and 20%, and consist almost entirely of hematite losses. Investigations show that grains finer than 30 μ are generally not at all concentrated. During 1953 recoveries have increased by use of rod-mill grinding, causing the production of slime during grinding to decrease, and better sorting arrangements, as well as more effective slime tables, have also improved this process. The conditions are, however, not nearly so good as in the concentration of magnetite. Hematite and magnetite ores, which can be kept apart in mining, should therefore not be mixed unnecessarily. It should often not be necessary to make both magnetic and gravity separation on the tailing before it is discarded.

The effort to find a better concentration method for the large deposits of siliceous hematite ores has

led to the testing of metallurgical methods. After very comprehensive experiments in the mid-1940's at Striberg,[18] it was found necessary to discard for the present the idea of transforming the hematite into a form suitable for magnetic separation by means of magnetizing roasting. The author is not prepared to judge whether direct production of sponge iron from lean lump ore with subsequent magnetic separation would be more profitable; the future of hematite concentration may lie in flotation, even if there may be present difficulties in sintering the fine hematite concentrate obtained by flotation.

In the 1940's Bring made important experiments on the development of hematite flotation, and since then the principles of oxide-ore beneficiation have greatly developed.[10–13]

To study further the possibility of flotation, experiments were made on Stripa and Striberg ores in the laboratories of the Department of Mineral Dressing at the Royal Institute of Technology, Stockholm. The following table shows a typical result:

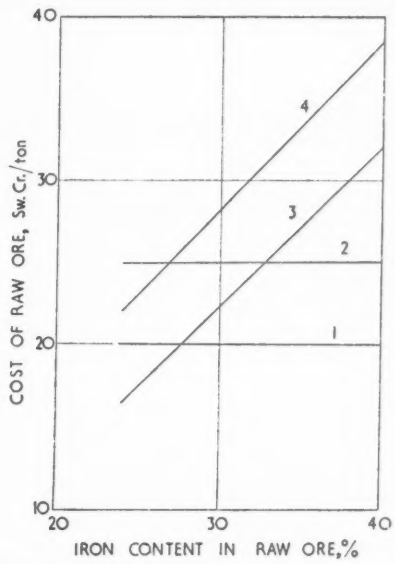
	Weight, %	Fe Content, %	Fe Recovery, %
Head	100	47·0	100·0
Concentrate	64·9	67·0	92·3
Middling	13·4	25·4	7·2
Tailing	21·7	0·99	0·5

In the continuous process the metal content of the middling is divided between concentrate and tailing. Comparison was made between the economic results of gravity separation and of flotation of a 67% concentrate, the value of which was assumed to be Sw.Cr. 68 per ton at the mine. The following assumptions were made:

	Gravity Separation	Flotation
Fe content of tailing in treating 30% ore, %	12	4
Fe content of tailing in treating 40% ore, %	16	5
Total cost, Sw.Cr. per ton of ore	20	25

Figure 13 shows that the economic margin obtained in flotation, as compared with gravity separation, is Sw.Cr. 0·80 per ton for 30% ore and Sw.Cr. 1·40 per ton for 40% ore. At a value of the concentrate of about Sw.Cr. 55 per ton, the methods would be economically equal under the above assumptions. This calculation is only meant to show that flotation of hematite ores should not be considered impracticable. Much work, however, remains to be done before definite calculations can be made, especially on the cost of reagents and the most favourable flotation conditions.

In magnetic separation iron losses are not at present high. Yields are generally above 95% and the contents of magnetic iron in tailings after crushing to liberation are often as low as 1–2%, thanks to gradual improvements in magnetic separators, several types of which now work almost without losses.



(1) Total cost and (3) value of concentrate per ton of raw ore in gravity concentration
(2) Total cost and (4) value of concentrate per ton of raw ore in flotation

Fig. 13—Economic comparison between flotation and gravity concentration of hematite ores; preliminary estimates of values, costs, and yields

Acknowledgments

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Reduction of Iron Ore Without Melting in a Rotary Furnace

By Bo Kalling and Folke Johansson

SYNOPSIS

This paper deals with a new method for reducing iron ores, without melting, in a rotating furnace. Tests have been carried out in a semi-scale furnace at the Domnarfvet Steelworks in Sweden. The construction of the furnace and the results so far obtained are described. Several ores have been tried as well as different kinds of fuel. A furnace of special type has been constructed for desulphurization of the sponge iron produced. The possibilities of the process for steelmaking are discussed. 928

Introduction

SPONGE iron for steelmaking is today produced on a commercial scale in several steelworks by the well-known Sieurin–Höganäs and Wiberg–Söderfors processes. At present it is mainly in Sweden that these methods have come into use ; this is principally due to the increased demand for a raw material low in phosphorus and sulphur to replace the increasingly expensive charcoal pig iron for quality steel production and especially for the acid steel processes. A contributory reason for the interest in sponge iron in Sweden is the increasing difficulty of obtaining sufficient quantities of scrap free from alloying elements such as nickel, chromium, and copper for the electric steel furnaces.

Recently, greater attention has been paid to the possibility of utilizing sponge iron to obtain a general decrease in the costs of steel production, including that of commercial steels. Sponge-iron processes can be carried out with better heat economy than the blast-furnace process, and in some cases much cheaper reducing agents than the usual blast-furnace coke can be used.

In this paper a short report is given of results of experiments made at Domnarfvet in recent years, where a method for reduction in a rotary kiln has been developed. The primary object of this work was not to make an especially pure product for quality steel production, but to find a cheap method of reduction.

The rotary kiln has often been considered in the past and tested for iron-ore reduction. Valuable work in this field has been carried out for many years by the U.S. Bureau of Mines,[1, 2] where a method was developed by which the ore is mixed with fine-grained coal and heated when passing the kiln by combustion gases from a gas burner inserted through the discharge end of the furnace. Interesting experimental results have been reported, but these do not seem to have been applied in commercial production. A similar method appears to be used commercially by the Anaconda Copper Mining Company[3] for the partial reduction of pyrite ashes to an iron powder which is later used for cementation of copper. In Norway a rotary kiln is also used by AS Titania for the partial reduction of an ore high in titanic acid, principally for the recovery of titanium.

In Sweden extensive experimental work was done on reduction in a rotary kiln at the Avesta Iron and Steel Works during 1928–1935.[4, 5] The heat required was supplied by an electric current led through the charge between two ring-shaped metal electrodes placed within the furnace. This method has not come into commercial use, although the kiln at Avesta was used for some time in the production of iron powder (*see* Fig. 4).

The limited success of the methods based on rotary kilns must primarily be due to difficulties in preventing the ore burden from balling in the furnace during reduction or from forming ring-shaped coatings on the furnace walls in the hottest zone ; the latter seems to be the more difficult to avoid. There is a very narrow margin between the temperature required for a sufficiently rapid and complete reduction of the ore and that at which sticking of the ore during reduction, usually intensified by the ash content of the fuel, tends to occur.

If the heat is supplied by the combustion of fuel within the furnace above the burden, a considerably higher temperature must be maintained in the furnace chamber and at the furnace walls above the burden than within the burden itself to secure a sufficiently rapid heat transfer ; this means a further decrease of the margin within which the process is practicable. This difficulty becomes more pronounced the higher the production, and many experiments may have failed because the maximum production under otherwise satisfactory technical conditions was too low.

With heating by an electric current within the burden, as at Avesta, it is possible to avoid overheating of the furnace chamber and thus to eliminate

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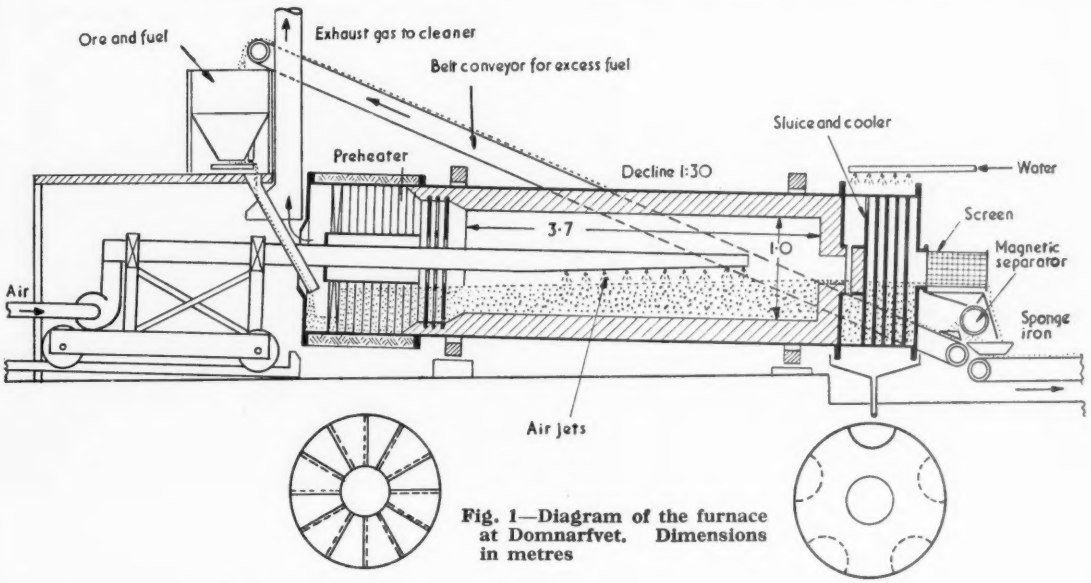

Fig. 1—Diagram of the furnace at Domnarfvet. Dimensions in metres

the formation of rings on the furnace walls. In this case, however, it is difficult to avoid the formation of balls in the charge at the current density through the material that is necessary for satisfactory production. It was found to be particularly difficult to avoid local overheating at the point of contact between electrode and burden and the resultant formation of coatings on the electrodes.

PRINCIPLE OF THE DOMNARFVET METHOD

The process developed at Domnarfvet differs from others using rotating furnaces mainly in the method of heat supply. As in the other methods, the burden consists of a mixture of ore and fine-grained coke or coal. The coke serves not only as a reducing agent but also as fuel for the process, so that a large excess of fuel must be used in circulation and a considerable part must be of very fine grain size. An essential feature of the process is that the air required for combustion of the coke and of the carbon monoxide formed during reduction is introduced through a central pipe inserted into the furnace. From this the air is blown through small tuyeres spaced along the pipe and directed towards the surface of the charge in the reduction zone. A diagram of the pilot plant at Domnarfvet is given in Fig. 1.

The most important advantage of this method of combustion is that the heat is developed on the surface of the charge or in its immediate vicinity. A considerable excess temperature results on that part of the surface which is in the combustion zone, but as the surface of the charge is continuously moving in the rotating furnace the overheated layer of the surface is rapidly mixed with those below, so that a rapid heat transfer and a homogeneous temperature are obtained. This method of heating has made it possible to keep the temperature of the charge as high as that of the furnace wall, and this considerably diminishes the risk of sticking at the walls.

However, the prevention of the formation of rings does not only depend on the temperature conditions in the furnace ; it is also important to avoid an excess of oxygen at any point in the reduction zone. Some fine-grained, more or less reduced ore will inevitably swirl up in the gas phase or stick to the brick joints or other irregularities in the furnace wall. If this iron comes in contact with free oxygen it is immediately burnt, causing a heavy local increase in temperature and the formation of coatings on the furnace walls. When, as in the present method, the air is directed towards the charge, combustion takes place very rapidly and completely, so that no oxygen is present in the furnace atmosphere outside the combustion zone.

It might be expected that it would be difficult to obtain a sufficiently high degree of reduction of the product while free oxygen is blown towards the surface of the charge ; however, this is not the case and very complete reduction can be achieved.

It is of great importance for the economy of the process that the method of introducing the air makes it possible to burn the carbon monoxide practically to completion within the furnace, so that the full heat content of the fuel can be utilized for reduction.

PILOT PLANT

The first preliminary experiments on the method were made in 1939 with a small furnace of the Avesta Steelworks at Högfors. Although these experiments were successful they were not continued ; in 1950, however, they were resumed at Domnarfvet, where the method has been developed further and a pilot plant has been set up (*see* Figs. 1 and 2).

The furnace is continuously charged with a mixture of ore and fuel ; the latter should be present in great excess. The ore type most generally used has been fine-crushed sinter, with coke breeze as reducing agent.

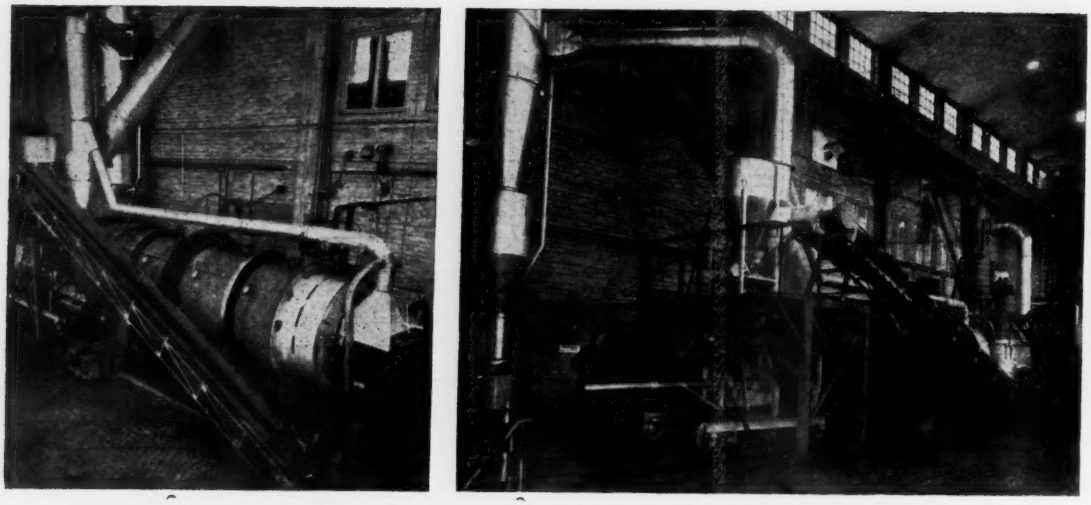

Fig. 2—Two views of the Domnarfvet furnace

The burden is fed into the reduction compartment *via* a heat exchanger, where it is preheated while the escaping reaction gas is effectively cooled. The heat exchanger consists of a number of transverse partitions of plate having large sector-shaped openings placed so that the gas is forced to alter direction when passing each partition. The three plates nearest the furnace chamber are cast from heat-resisting steel. To obtain a satisfactory level of the material in the heat exchanger the plate nearest the charging end is shaped as a shovel wheel.

The blast pipe is an essential detail of the construction. It is inserted from the charging end and rests on a carriage outside the furnaces, thus permitting withdrawal of the pipe for purposes of inspection. Along the pipe there are a large number of openings through which the air stream is directed towards the surface of the charge. The openings are arranged along the blast pipe in such a way that no surplus of oxygen should exist in any part of the reduction zone.

The treated material is removed from the furnace through a water-cooled sluice, which at the same time prevents the gas from escaping the same way.

At the discharge end the material is first passed over a screen, where it is divided into two fractions, above and below about 3 mm. The coarse material then consists only of sponge iron and need not be separated. The fine material, consisting of a mixture of sponge and excess fuel, is passed over a magnetic separator which selects the fine sponge ; the coke is carried back to the charging end by means of a conveyor belt.

The combustion gases, which normally contain a considerable amount of fine coke breeze, are purified in a cyclone ; most of the dust is separated by this and can be returned to the process.

The furnace chamber has an inside diameter of only 1000 mm. and its length is 3700 mm. It is lined with ordinary fireclay bricks ; the strength of the lining has proved very satisfactory. The decline of the furnace is 3·6% and the speed of rotation about 4 r.p.m.

GENERAL OPERATIONAL RESULTS

Ore

A large number of ores have been tried, with different pretreatments. Very fine-grained ores are not suitable for the process, at least in its present form, as the finest fraction (of grain size below about 0·05 mm.) swirls up in the furnace chamber because of the rotation and air blast, and therefore tends to stick to the furnace wall even if the furnace atmosphere outside the combustion zone contains no excess oxygen. However, it has also been possible to reduce unsintered ore concentrates, although better results are obtained if the finest fraction is removed by screening.

The best results have been obtained with ordinary sinter, which was crushed before charging to below about 15 mm. However, ore grain size and reducibility are not the only factors influencing the process. It has been found more difficult to obtain a satisfactory degree of reduction with pellets than with pan sinter, although on a laboratory scale the former show better reducibility. One reason is that pellets, consisting mainly of Fe_2O_3, have lower strength during reduction than ordinary sinter which, at least in the hard-burnt form that has been found to be the best, is far less completely oxidized. In addition, the powder obtained when the pellets disintegrate is much finer because a finer-grained concentrate is used for pellets than in other sintering methods.

The condition of the ore appears to have a further effect on the course of the process. If the ore is heavy and is available in round or rounded pieces, the burden seems to become more compact, the coke breeze is forced away, and the necessary intimate contact between ore and reducing agent is not obtained. The lighter pan sinter with its porous grains of irregular form is much better in this respect.

Some lump ores have also been tried, mostly with good results. The suitability of an ore depends

primarily on its composition. Magnetites are in general preferable to hematites although they are more difficult to reduce, as they do not disintegrate so much during reduction. The suitability of the ore also depends on the composition of the gangue, which in some cases is so easily melted that reduction becomes difficult or impossible.

Lump ores have in several cases given better reduction results than pellets, which may seem strange as the specific weight of the ore is generally higher. The grains of the lump ore, however, are often flatter than those of the pellets, which is an advantage, and they do not break up to the same extent and to such a fine powder as the pellets.

Poor lump ores can also be reduced to sponge iron of a high degree of reduction. By crushing and concentrating after reduction it is often possible to obtain a very pure product so that, when ores difficult to grind and to concentrate are to be reduced, it may be more advantageous to carry out the fine crushing and the concentration after the reduction and avoid pretreatment of the ore.

Fuel and Reducing Agent

In most tests coke breeze was used as reducing agent. The grain size should in general be below 2 mm. It is essential that the coke is not too coarse, as its reactivity with the ore as well as with the oxygen in the air decreases rapidly with increased grain size. If it is too coarse, therefore, the degree of reduction will tend to be low, and there will be poor combustion and an excess of oxygen with the consequent risk of sticking. If, on the other hand, the coke is too fine, coke consumption is increased because of the excessive CO content in the waste gas and greater dust losses. The fraction below 0·1 mm. seems mainly to follow the exhaust gases from the furnace to the cyclone. It is very important to choose a coke with the right grain size when planning the process, and as a means of adjustment in this respect it is preferable to have two qualities of coke with different grain sizes available.

The fine-grained coke breeze that was used in the authors' last experiments was obtained by screening the fraction below 2 mm. from the breeze delivered from a blast-furnace coke screening plant. This method, however, is not to be recommended, as this fraction will contain most of the impurities. All the coke breeze should be crushed down to the required grain size, but in such a way that a minimum quantity of fines is obtained. Crushing in a rolling crusher seems to give a suitable grain distribution.

The composition of the coke breeze can affect the course of the process considerably. In most coke qualities the coke ash does not begin to melt until about 1275° C., but the iron oxides present may cause melting or sintering phenomena at far lower temperatures. However, agglomeration that can be directly attributed to the quality of the coke has seldom been observed at normal reduction temperatures, *i.e.*, 1050–1100° C. A temperature of 1100° C. in the hottest zone of the furnace is generally sufficient even for ores that are difficult to reduce.

If the screen analysis of the coke and the distribution of the combustion air are suitably adjusted, there is no difficulty in burning the carbon monoxide within the furnace chamber practically to completion. In these experiments it has, however, proved advantageous to regulate the run so that the exhaust gas contains 2–3% of unburnt carbon monoxide ; the content of carbon dioxide will normally be 28–30%. In spite of the comparatively complete combustion of the gas it has been possible to keep the temperature of the exhaust gases as low as about 250° C.

Random samples of the gas from the end of the reduction zone immediately after the last air outlet have generally given a CO_2 content of about 20% and a CO content of 10–15%. Although gas of that composition has an oxidizing effect on sponge iron, degrees of reduction of up to 95% and even more have been obtained ; this is good evidence of the possibility of reducing in an oxidizing gas atmosphere in the furnace chamber in the presence of carbon in the charge.

Fuels other than coke breeze can be used with advantage in the process ; for instance, breeze of anthracite of about the same grain size as coke breeze has given excellent results. The ash of the breeze used had a somewhat higher melting point than that of ordinary coke breeze. Coal powder has also been tried, but it is difficult to utilize the heat value of the distillation gases satisfactorily in the process. In this case it is better to carry out a low-temperature coking in a special kiln before using the fuel in the sponge-iron furnace, and to collect the distillation product of this process. An excellent reducing agent is obtained in this way. This coke need not be very strong but it must not have too strong a tendency to break up into a fine powder in the reduction furnace. In the coking process it may be convenient to add the dust collected in the cyclone.

Oil or other lighter hydrocarbons may also be added to the process in small quantities to facilitate the reduction, especially if the reactivity of the solid fuel has been found to be too low, *e.g.*, because of too large a grain size. The oil should preferably be introduced from the discharging end.

Oxygen can at least partially be substituted for air with very good results, but to run the process continuously with oxygen or oxygen-enriched air does not seem to give advantages that will compensate for the increased cost. The use of oxygen has, however, been found valuable for increasing the temperature rapidly if desired.

Production Capacity

Normal production rate with a sinter charge containing 60% Fe has been about 3 tons of iron per day. As the charge volume is about 1 cu. m. this corresponds to about 3 tons per cu. m. per day. On the basis of total furnace volume the production has been 1 ton of iron per cu. m. per day.

The normal time for the ore to pass through the reduction chamber of the furnace is 5–6 hr.; it is at the full reduction temperature for about half of this period. This time seems to be quite sufficient for particles up to 15 mm. or even larger. It is of interest that the coarser particles are normally just as well reduced as the smaller ones ; this is attributed to the fact that during treatment the ore is in contact

alternately with a reducing and an oxidizing atmosphere.

The coke is not as heavy as the ore, so that it is partly blown away from the ore in the air-blast zone and becomes concentrated in a layer close to the wall. This has in general caused no trouble, but the non-uniformity of the charge should naturally not become excessive. The air supply must therefore be kept below a certain value, and this will influence the maximum production rate.

Coke Circulation

The total quantity of coke charged together with the ore has normally amounted to about twice the quantity consumed, so that the weight of circulating coke has been about the same as that of fresh coke added. However, the excess of coke in the furnace is much smaller than that corresponding to the proportions between the charged quantities of ore and coke, because the coke passes more rapidly through the central discharge opening than the ore, which is heavier and also considerably coarser.

An important condition for the economy of the process is that the coke ash does not follow the return coke. Continuous analysis has shown that the ash content of the return coke amounts to only a few per cent. more than that of the fresh coke, even after continuous operation has been reached, and that the ash content is often quite unchanged. Nor is the reducing capacity of the return coke diminished, so that it can be utilized completely in the process.

According to the analyses, most of the coke ash leaves the furnace together with the exhaust gases. Its grain size is so fine that it does not even remain in the cyclone but most of it disappears with the stack gases. A certain part of the coke ash sticks to the finer fraction (<3 mm.) of the sponge iron, but the coarse sponge always remains clean. The quantity that will stick to the sponge depends on the composition of the coke breeze and the gangue, and also on the reduction temperature. Probably not more than 10% of the coke ash will accompany the sponge iron, 5% is found in the cyclone dust, and the rest of the ash disappears in the stack. In tests with a comparatively low temperature of reduction it was found that even the fine-grained sponge was completely free from coke ash. If a fuel with ash of a higher melting point than that of coke breeze is used, it is probable that no ash will be present as impurity in the sponge.

The dust in the exhaust gas contains an important part of the coke charged. Most of this coke dust is deposited in the cyclone. The dust content of the exhaust gas depends in the first place on the grain size of the coke used, and is also increased a little if the air supply is increased. In the most recent test periods, when accurate weighings were carried out, the coke content of the cyclone dust amounted to not less than about 15–20% of the total coke consumption. This quantity can no doubt be reduced considerably by using a coke breeze containing less fines, but, on the other hand, the coke must probably contain a certain quantity of dust-fine material to facilitate combustion in the furnace.

When ordinary sinter was used, the cyclone dust contained, besides coke breeze, about 15% of iron oxides as well as a small percentage of coke ash. The iron content amounted to 1–1·5% of the quantity of concentrates charged. In the case of pellets the iron losses amounted to three times as much, owing to the far smaller grain size of the original concentrates in this case.

In some tests the cyclone dust was allowed to circulate in the process by adding it to the materials charged. This does not seem to be a disadvantage ; on the contrary, the increased quantity of fines in the furnace seems to be favourable for the process. However, it has not been possible to utilize the cyclone dust completely in this way ; for that purpose the breeze should perhaps be added to the burden further inside the furnace, possibly from the discharging end.

Fuel Consumption

On the basis of the quantity of air supplied and the analyses of the exhaust gases it has been possible to calculate exactly the quantity of coke that is actually used in the furnace by reaction with oxygen in air and ore. The remaining carbon content of the coke charged is distributed between the sponge iron, the cyclone dust, and the exhaust gases. The distribution of carbon was calculated for a high (2800 kg. of iron per day) and for a low (2200 kg. of iron per day) production level at a time when the quantity of cyclone dust was comparatively low. The results were as follows :

	Carbon Distribution, kg. per ton of iron	
	High Production	Low Production
Carbon burnt	392	432
Carbon in exhaust gases (mainly cyclone dust)	96	72
Carbon in sponge iron	12	12
Total	500	516
Corresponding quantity of coke, assuming 80% C content	625	645

If the process were carried out in a full-scale furnace, a decrease in burnt carbon of about 50 kg. per ton of iron would probably result, because of lower heat losses. It should also be possible without difficulty to diminish the dust losses to about half of those mentioned. If these conditions are fulfilled the consumption of coke breeze containing 80% of carbon will only be about 500 kg. per ton of iron.

The possibility of such a coke consumption is proved by the theoretical calculation for the process given in the Appendix.

Composition of the Sponge Iron

A degree of reduction of 85–95%, depending on the reducibility of the ore, has been attained. As mentioned above, it is as high in the coarse sponge as in the fine.

The carbon content of the sponge iron varies considerably about an average value of 1%. Generally this carbon is largely mechanically mixed with the product and, with fine-grinding and magnetic concentration of the sponge, its content can be reduced to some tenths of 1%.

The phosphorus content of the sponge is generally somewhat higher than that of the ore, as phosphorus from the coke ash is absorbed by the sponge iron. However, the increase in phosphorus content is only 0·010–0·015%, which is of no importance if the subsequent steel melting takes place in a basic furnace.

The sulphur absorption from the coke during the reduction, on the other hand, is considerable and this is a disadvantage. The quantity absorbed by the sponge varies and depends in the first place on the analysis of the ore. With ores having a gangue consisting entirely of silicic acid or silicates, the absorption of sulphur was generally about 2·5 kg. per ton of iron in the coarse fraction of the sponge and somewhat higher in fractions below 3 mm. This means that about 60% of the coke sulphur goes over to the sponge iron, whereas the remaining 40% disappears with the exhaust gases. The sulphur content of the sponge will be higher if ores containing lime are treated.

Some attempts were made to counteract the absorption of sulphur in the sponge by adding granular limestone, burnt lime, or dolomite to the charge. However, the added material disturbed the process, since it reacted with the coke ash and the gangue of the ore to form comparatively low-melting silicates which caused ring formation on the furnace walls at the temperature necessary for reduction. These tests are still proceeding.

It may be more advantageous to desulphurize after reduction. The sponge iron may be desulphurized in solid form before melting, but in that case it must be fine-crushed before treatment. In a method that had already been tested at the Avesta Steelworks[4, 5] the sponge is heated to 800–850° C. in a rotating furnace together with lime and some coke breeze. As the presence of oxidizing gases must be prevented, electric heating should be used. After treatment the sponge is separated from the lime, which now contains the sulphur, by magnetic separation.

Desulphurization of the sponge iron by this method has been tried in a laboratory furnace in which effective heat exchange between the incoming and outgoing material is possible. As this type of furnace can also be used for the heat-treatment of other materials in granular or powder form, some information about its construction and the results obtained will be given.

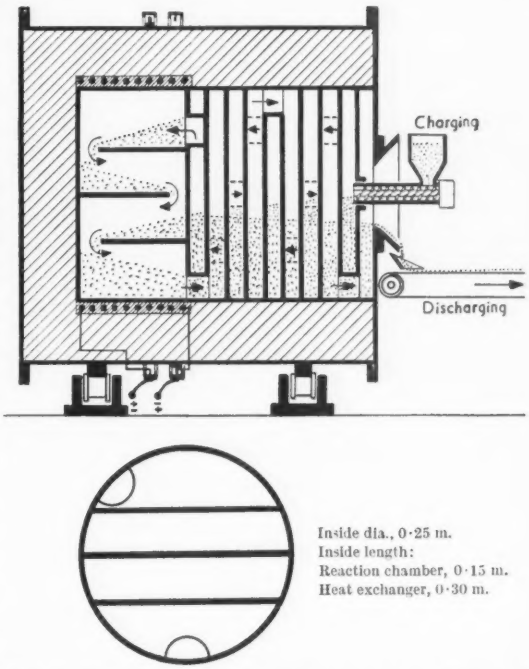

Fig. 3—Diagram of small-scale desulphurizing furnace

The principle of the recuperative furnace and its main dimensions are shown in Fig. 3. The heat exchanger is divided into a number of small compartments by transverse partitions, of which alternate pairs are connected by an opening along the periphery. Of the two systems thus obtained, one is used for charging and one for discharging of the material which, owing to the difference in level, flows into and back through the rotating furnace.

The reaction chamber is electrically heated by resistance wiring around the mantle. The charge is fed into the first compartment of the heat exchanger by means of a screw by which the feeding rate can be adjusted.

Table I

RESULTS OF REDUCTION OF DIFFERENT ORES

Ore Type	Constituent	Degree of Oxidation, %	Degree of Reduction, %	Analysis, %													Reduction Temp., °C.	Production, kg./hr.
				Total Fe	MnO	MgO	CaO	Al_2O_3	SiO_2	TiO_2	P	S	C	Zn	Pb		
Sinter of pyrite ash	Sinter	92·2		64·4	...	1·07	1·48	1·14	3·90	...	0·013	0·40	–	0·36	0·08	1050	114
	Sponge iron		91·4	81·5	...	2·62	2·54	1·74	7·70	...	0·010	0·64	0·56	0·03	Tr.		
Striberg ore in small lumps	Ore	96·0		55·4	0·13	0·60	2·05	0·98	18·0	...	0·017	0·003	–	–	–	1070	110
	Sponge iron		84·5	67·4	0·10	0·78	2·60	2·02	23·1	...	0·028	0·212	1·55	–	–		
Sinter of Vintjärn concentrate	Sinter	93·6		59·6	1·37	3·76	2·52	1·43	6·90	0·12	0·008	0·010	–	–	–	1070	120
	Sponge iron		85·6	73·8	1·52	4·37	3·20	1·72	8·28	0·18	0·016	0·218	1·10	–	–		
Sinter of Grängesberg concentrate	Sinter	92·3		68·2	0·28	0·72	0·82	0·57	1·80	0·13	0·022	0·003	–	–	–	1095	90
	Sponge iron		90·9	89·7	...	0·76	0·76	1·24	3·80	...	0·028	0·251	0·85	–	–		

Table II

RESULTS OF OPERATION WITH VINTJÄRN ORE SINTER

Test Period	1	2	3	4
Time of observations, hr.	31	36	95	39
Charged Materials				
Sinter, kg./hr.	186·1	198·5	151·0	150·1
Coke breeze, kg./hr.	83·1	85·7	68·9	61·8
Return coke, kg./hr.	66·9	84·3	71·1	68·2
Dust from cyclone, kg./hr.	10·3	—	—	20·0
Blast, excluding moisture, cu. m. at N.T.P./hr.	235	247	214	211
Discharged Materials				
Sponge iron, kg./hr.	146·4	157·8	118·4	117·7
Return coke, kg./hr.	66·9	84·3	71·1	68·2
Dust from cyclone, kg./hr.	31·3	21·8	15·8	29·0
Exhaust gases, cu. m. at N.T.P./hr.	268	283	241	237
Analyses				
Sinter*: Fe, %	61·1	61·1	59·6	58·9
Sponge iron*: total Fe, %	76·4	75·5	75·0	74·4
reduction, %	84·1	81·8	85·0	83·6
carbon, %	1·01	1·64	1·10	0·88
Moisture in coke breeze, %	3·5	6·4	5·9	6·7
Dry coke breeze: carbon, %	79·4	80·2	78·1	77·2
ash, %	14·2	14·4	13·6	14·1
Fe, %	1·8	2·0	1·6	1·5
Return coke: ash, %	24·6	19·0	15·8	16·4
Dust from cyclone: carbon, %	63·0	66·4	67·0	63·6
ash, %	33·3	30·7	27·2	29·9
Fe, %	11·0	12·6	9·3	10·3
Exhaust gases: CO_2, %	25·9	26·2	25·6	25·8
CO, %	4·7	4·8	4·3	3·8
Production: total Fe in sponge, kg./hr.	111·9	119·2	88·8	87·6
Yield of Fe, %	98·4	98·3	98·7	99·1
Consumption of coke, calc. on dry breeze, kg./hr.	80·2	80·2	64·8	57·7
Ditto per 1000 kg. total Fe in sponge, kg.	717	673	730	659
This corresponds to carbon, kg./ton Fe	569	540	570	509
of which: carbon burnt, kg./ton Fe	392	394	435	429
carbon in sponge iron, kg./ton Fe	13	22	15	12
carbon in cyclone dust, kg./ton Fe	118	122	120	65
balance, kg./ton Fe	46	2	—	3
Reduction temperature, ° C.	1085	1070	1070	1065
Temperature of materials leaving heat exchanger, ° C.	644	677	668	648
Temperature of exhaust gases, ° C.	263	246	220	218

*Further analyses are given in Table I

In this furnace, sponge iron ground to <1·5 mm. and containing 90% Fe was treated at 840° C. with 3% of lime and 6% of coke. In this way the sulphur content was decreased from 0·24 to 0·03%; the production rate was 7·5 kg. of sponge per hr. The power consumption was 1·43 kW. and the temperature of the discharged material was 190° C.

The energy consumption in these tests can be divided as follows:

	kcal./hr.	kWh. per ton of sponge
Heat content of discharged material	179	28
Heat of reaction	17	3
Heat losses (balance)	1034	160
Total	1230	191

Heat losses in this small laboratory furnace, when calculated per ton of sponge iron, are naturally disproportionately high. For a larger furnace treating 1 ton of sponge per hr. it should easily be possible to reduce them to 60 kW., in which case the total consumption of energy would be only 90 kWh. per ton of sponge, or 100 kWh. per ton of Fe.

It may be more economical, however, to combine the desulphurizing of the sponge iron with the subsequent melting of the product. If a basic slag is used, it will be easy to obtain an adequate desulphurization. The melting should preferably be carried out in a simple open electric low-shaft furnace; a sufficient quantity of lime and of some coke should be added to bring the reduction to an end and to give the molten iron the carbon content necessary for the subsequent steelmaking process. The carbon content can be varied from about 1% up to normal pig-iron content. If the production of pig iron along these lines is contemplated, it might be more suitable to carry out the melting by using a comparatively acid slag and to desulphurize the pig iron after melting.

RESULTS OBTAINED

Reduction tests have been made with a large number of ores, and some detailed results from different experimental periods are given in Tables I–III.

One type of ore that has proved to be very easy to reduce and which has favourable physical properties is sinter made of pyrite ashes. Degrees of reduction of up to 95% have easily been obtained with a temperature in the hottest zone of about 1050° C. Table I gives analyses of average samples, of the sinter as well as of the sponge obtained, taken during a period of operation of 5 days. The somewhat high contents of

Table III

SCREEN ANALYSES OF FUEL AND CYCLONE DUST

Material	Fraction, %										
	–0·06mm.	–0·075mm.	–0·10mm.	–0·15mm.	–0·20mm.	–0·30mm.	–0·50mm.	–0·75mm.	–1·0mm.	–1·5mm.	–2·0mm.
Coke breeze	3·4	6·5	9·7	15·0	21·4	30·4	49·5	65·3	73·7	93·4	98·5
Return coke	1·3	2·6	4·1	14·6	25·1	40·2	60·8	79·0	90·0	97·0	99·7
Cyclone dust	91·6	95·0	96·4	98·2	99·4	100					

CaO and MgO in the sponge result from the fact that the sintering of the pyrite ash was carried out with a rather poor bedding ore which could not be removed completely in sampling. The unusually high sulphur content of the sponge originates from the high sulphur content of the sinter. Almost complete distillation of zinc and lead has taken place during the reduction ; if the contents of these elements are considerable it may be worth collecting the oxides of these metals in the exhaust gases.

The Striberg ore included in Table I consists of a quartz-banded hematite very difficult to concentrate ; this was charged in lumps of 3–10 mm. As a large proportion of the iron oxides is intimately mixed with the silica, it is rather difficult to reduce this ore, but a degree of reduction of 85% could nevertheless be attained without trouble. The sponge iron was afterwards finely ground ; it was found that the grinding was much easier with the sponge than with the raw ore. By subsequent magnetic separation a sponge was obtained with a content of 92% Fe and a degree of reduction of 90%. The iron recovery amounted to 91%, a value far higher than that obtained by concentration of the ore before reduction.

The comparatively poor sinter from Vintjärn included in Table I and used in most of the latest tests was also difficult to reduce and the degree of reduction could not reach higher average values than about 85%. With the rich Grängesberg sinter, however, a degree of reduction above 90% could easily be attained.

The tests with pellets and unsintered fine-grained concentrates have so far, for reasons mentioned above, not given satisfactory results, at least during longer periods of operation.

During some recent periods of experiments with Vintjärn sinter observations were made to determine in detail the material balance and other conditions during the operation. The results from four such periods are given in Table II.

The screen analyses of the coke breeze used in these tests varied considerably. Those shown in Table III refer to period 4 in Table II, when reducing conditions were good and dust losses relatively small.

APPLICATIONS AND FURTHER DEVELOPMENT

As the method has so far not been tried on a commercial scale, it is impossible to pronounce with any certainty upon its future. It can be expected, however, that the interior diameter of the furnace can be increased from 1·0 to 2·0 m. and the length from 3·7 to 6 m. without any unexpected operational diffi-

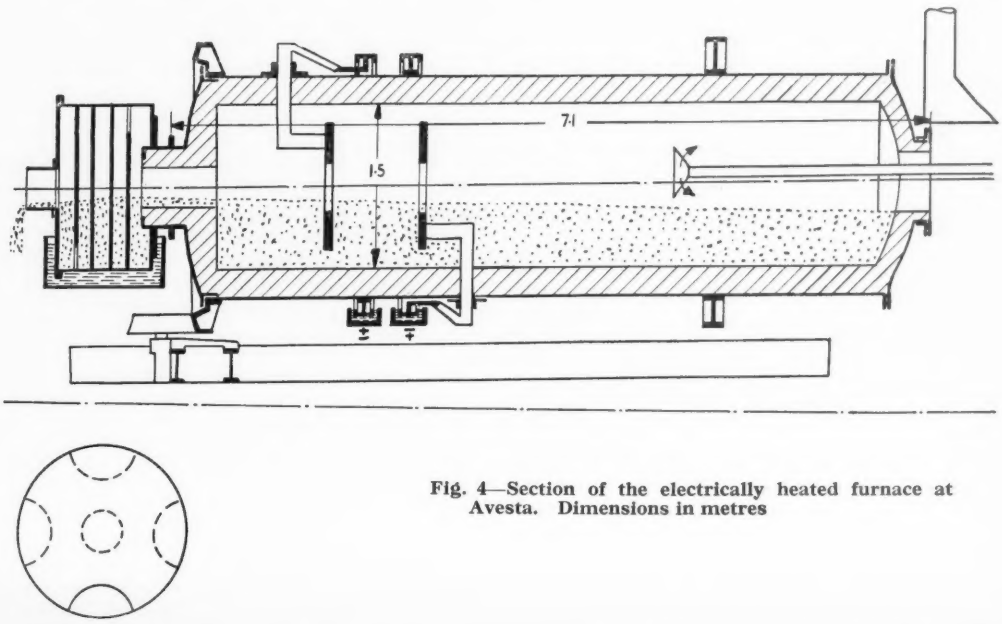

Fig. 4—Section of the electrically heated furnace at Avesta. Dimensions in metres

Table IV

MATERIAL BALANCE FOR REDUCTION OF IRON ORE

Air Blast and Exhaust Gases

Reaction	Carbon, kg.	Oxygen, cu. m. at N.T.P.	Air Blast, cu. m. at N.T.P.	Waste Gases, cu. m. at N.T.P.		
				CO_2	N_2	Water
1	35·8	—	—	67	—	...
2	96·7	—	—	180	—	...
3	175·5	328	1562	328	1234	...
Totals	308	328	1562	575	1234	25

Charged Materials

Material	Fe_3O_4, kg.	Carbon, kg.	Ash + Gangue, kg.	Water, kg.	Total, kg.
Ore	1382	—	47	—	1429
Fresh coke	—	320	60	20	400
Return coke	—	480	120	—	600
Totals	1382	800	227	20	2429

Discharged Materials

Material	Fe, kg.	FeO, kg.	Carbon, kg.	Ash, kg.	Total, kg.
Sponge iron	900	129	12	50	1091
Return coke	—	—	480	120	600
Coke ash in exhaust gases	—	—	—	57	57
Totals	900	129	492	227	1748

Table V

HEAT BALANCE FOR REDUCTION OF IRON ORE

Heat Input	Mcal./ton total Fe	
Combustion of 175·5 kg. of carbon (reaction 3)		1379
Heat Output		
Reduction of 1000 kg. Fe (reaction 1)	154	
„ „ 900 kg. Fe (reaction 2)	280	
Vaporization of 20 kg. of water	12	
		446
Heat content at 1050° C. of:		
900 kg. Fe	153	
129 kg. FeO	25	
492 kg. C	184	
170 kg. of gangue and ash	46	
		408
Heat content at 250° C. of:		
57 kg. of ash	3	
575 cu. m. at N.T.P. of CO_2	58	
1234 cu. m. at N.T.P. of N_2	85	
25 cu. m. at N.T.P. of H_2O	2	
		148
Radiation losses		377
Total		1379

culties. By these steps the furnace volume would be increased 6·5 times, and a production of at least 15 tons per day should be attained. However, it is questionable whether still larger furnaces should be erected. It might be better to carry out the process in a larger number of comparatively small units so arranged that the charging, separation, and discharging can be combined for all furnaces. The erection costs for such a plant would be fairly low, and so would the labour costs, because of the fully continuous operation.

The greatest advantage of the method is the low consumption of fuel and the fact that cheap qualities of coal can be used provided that the ash does not have too low a melting point.

The disadvantage mentioned above, that the method in its present form is not suitable for reduction of very fine-grained ores, naturally restricts its field of utilization. More favourable results would be obtained in this respect if the air supply, particularly in the final reduction zone, could be reduced so that the finest ore powder did not tend to swirl up in the furnace chamber and to stick to the walls to the same extent. This could be arranged by an electric current supply in the final reduction zone, so that the air supply in that zone could be dispensed with entirely ; at the same time it would be possible to keep the atmosphere less oxidizing, so that the risk of sticking to the walls would be eliminated. It would then also be possible to attain a higher degree of reduction in the product. A power supply of only 100–200 kWh. per ton of iron would probably result in a definite improvement in this respect.

The supply of electric current can be arranged in various ways. The method used at Avesta seems to be the most suitable; Fig. 4 shows a longitudinal section of the Avesta kiln.

The possible production per furnace unit depends on the degree of reduction required. The production is increased considerably if the requirements in this respect are modified. Under certain conditions, therefore, it may be better not to carry the reduction to completion in the rotating furnace but only to pre-reduce the ore to a suitable degree of reduction and to carry out the final reduction in connection with the melting, *e.g.*, in electric low-shaft furnaces of the ordinary type. In this way considerable savings in power can be achieved in the production of electric pig iron. The degree to which the ore should be pre-reduced in the rotating furnace must be decided in each individual case.

No calculation of costs will be presented here, because the method has not yet been tried commercially and because the costs depend so much on local conditions that a general calculation would be of little value. As a general conclusion it may be said that the method makes possible the production of a very cheap iron, and it therefore merits consideration in comparison with other methods for the reduction of iron ore, especially when erecting new plants where the production methods may still be chosen freely.

Acknowledgments

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APPENDIX

Heat Balance for the Reduction Process

The following heat balance is based on a 90% degree of reduction of a magnetite ore containing 70% of iron and 3·3% of gangue.

Coke breeze containing 80% of carbon, 15% of ash, and 5% of water is used as fuel. The total amount of fresh coke and return coke is 1000 kg./ton of iron. The return coke analysis is 80% of carbon and 20% of ash, and 15 kg. of coke of this composition are assumed to remain in the sponge iron.

The reduction is assumed to take place at a final temperature of 1050° C. and the reduction gas as well as the coke is burnt to 100% CO_2 and N_2. The exhaust gas is used for preheating the ore and coke, and leaves the furnace at 250° C. together with the water and the ash from the burnt coke. Air humidity is neglected.

On the basis of measurements on the experimental furnace, the heat losses for a furnace producing 15–20 tons of iron per day are assumed to be about 375 Mcal. per ton of iron. The total amount of coke necessary for reduction and heating will then be 385 kg. per ton of iron. Including the coke in the sponge iron but excluding dust losses, the total coke consumption will be 400 kg./ton of iron.

The final reactions are :

(1) $2\,Fe_3O_4 + C = 6\,FeO + CO_2$; $\Delta H = 51{\cdot}7$ kcal.
(2) $2\,FeO + C = 2\,Fe + CO_2$; $\Delta H = 34{\cdot}7$ kcal.
(3) $C + O_2 = CO_2$; $\Delta H = -94{\cdot}3$ kcal.

The material balance for the reduction of 1000 kg. Fe according to equation (1) and 900 kg. Fe according to equation (2) is given in Table IV, and Table V gives the heat balance for the process.

A Theory of Hydrogen Embrittlement

By F. de Kazinczy

SYNOPSIS

Hydrogen embrittlement is caused by a lowering of shear strength and cleavage strength. This can be explained by assuming that molecular hydrogen of high pressure is included in a Griffith crack or some other crack, which initiates fracturing. During crack spreading the gas expands and releases energy, which results in a lowering of the fracture stress. It is shown that hydrogen diffusion into the crack is needed during crack spreading, by which the time and temperature effect of hydrogen embrittlement can be explained.

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Introduction

THE relation between the hydrogen content and the strength and ductility of steel has been the object of many investigations. To review the effect of hydrogen, the most important of the results will be summarized here.

The Young's modulus is not changed by hydrogen[1] and the yield point is only lowered in some exceptional cases.[2–4] The hardness[5, 6] and maximum tensile stress remain unaffected by hydrogen in the investigated range up to 10 ml./100 g., but the ultimate tensile stress is strongly decreased.[3–5, 7–9] The elongation at fracture and the reduction in area first decrease with increasing hydrogen content, but above a certain value, which for unalloyed steel at room temperature seems to be about 5–7 ml./100 g., this effect diminishes rapidly, and ductility decreases slowly when hydrogen content is further increased. The shape of the true stress/strain curve remains unaffected, but rupture occurs at a lower stress and true reduction in area.[3, 5, 8] Increase of stress with unchanged strain hardening has been reported in only one case.[5] The effect was small, however, and was not accompanied by a corresponding increase in hardness, as would have been expected.

The impact value was in some cases reported to be unaffected,[4, 10] but a small lowering of it was found in a more careful investigation,[3] in which a raising of the transition temperature from the notch-brittle to the notch-ductile condition is also reported. Generally, it can be stated that hydrogen embrittlement decreases with increased speed of deformation above a certain value. This speed is estimated at room temperature to be that corresponding to a time of 10 sec. for fracture in a tensile test.[10]

With respect to the influence of temperature, it was reported[11] that hydrogen embrittlement decreased and almost disappeared below −110° C. In a very careful investigation[12] the reduction in area was examined as a function of hydrogen content at

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different temperatures for several tempered structures (*see* below). It was generally observed that the original ductility is regained when hydrogen is removed.

Before the mechanism by which hydrogen can cause embrittlement can be discussed, the way in which hydrogen is occluded in steel must be understood. The relation between hydrogen dissolved in the lattice (x_1 ml./100 g.), temperature (T° K.), and pressure (p atm.) has been given by Phragmén[13]:

$$T = \frac{3000 - 0{\cdot}062\,p}{\log p - 2\log x_1 + 3{\cdot}60} \quad \text{.....(1)}$$

The pressure of hydrogen gas in thermodynamic equilibrium with a given amount of dissolved hydrogen x_1 increases with decreasing temperature. For example, the hydrogen pressure for 5 ml./100 g. dissolved in the lattice at 600° C. is 17 atm.; at 300° C., 880 atm.; at 200° C., 4080 atm.; at 100° C., 10,870 atm.; and at 20° C., 17,500 atm. However, the pressure can never exceed 48,800 atm.

Cracks, cavities, and similar voids in steel are filled at equilibrium with hydrogen of a pressure according to equation (1) for the given values of x_1 and T. When hydrogen pressure in the cavities differs from the equilibrium pressure, gas will diffuse to or from the cavities. In this treatment any possible non-diffusible hydrogen will be disregarded.

To calculate the amount of hydrogen included in the cavities, the relative volume of the cavities has to be known. Up to 15,000 atm. the following approximate equation of state can be used for gaseous hydrogen:

$$\frac{pV}{1 + \frac{p}{p_a}} = nRT$$

where $p_a^{-1} = 0{\cdot}0006$. This gives the amount of gas contained in the cavities as:

$$x_2 = \frac{pv}{T} \cdot \frac{34{\cdot}8}{1 + 0{\cdot}0006\,p} \quad \text{.....(2)}$$

where x_2 is in ml./100 g., and v is the relative volume of cavities in vol.-%.

As well as the hydrogen dissolved in the lattice and included in cavities a large amount of hydrogen probably occurs around dislocations. This hydrogen behaves differently from that in cavities. It is carried along with migrating dislocations during plastic deformation; evolution of hydrogen at slip lines on the surface of the specimen has often been observed during plastic deformation. The amount of gas around dislocations will, however, not influence the equilibrium pressure or activity of the hydrogen, and in many cases it is useful to treat it as if it were also included in voids.

In early theories of hydrogen embrittlement, especially in many of Chaudron's papers, it was assumed that hydrogen dissolved in the lattice in some way could render slip more difficult, thus causing embrittlement. Later it was proposed that high gas pressure in the cavities causes triaxial stresses in their immediate surroundings, which renders slip more difficult and leads to the initiation of brittle failure at the cavity.[4-6, 9, 13, 14] The effect of high deformation speed and low temperature on hydrogen embrittlement has usually been explained by assuming that the cavities are first formed during plastic deformation, and that a temperature-dependent hydrogen diffusion is then needed to build up the gas pressure in these cavities.

All these theories fail to explain why hardness is not increased by hydrogen, as would be expected as a result of hindered slip. Another consequence should be that the yield point is raised by hydrogen, which is also contradictory to experimental results.

Recently it has become clear that hydrogen does not influence the resistance to slip; it only affects the U.T.S., the consequence of which is a lowering of the reduction in area and elongation at fracture. Petch[15] considered only cleavage and intercrystalline fractures, and suggested that hydrogen lowers the brittle strength, the intersection of the cleavage-strength/strain and the true-stress/strain curves giving the new U.T.S. He related the effect of hydrogen to the Griffith theory of brittle fracture, and proposed that an adsorbed layer of gas on the surface of the crack will lower the surface energy, thus leading to a lower cleavage strength. As the gas has to diffuse from the lattice to a clean crack surface, diffusion of hydrogen gives the time and temperature effect. But Petch does not explain how different hydrogen contents can give rise to different values of cleavage strength.

Bastien and Azou[8] suggested another mechanism of hydrogen embrittlement, also to explain a lowering of the U.T.S. According to their theory, hydrogen is mainly concentrated around dislocations, and there forms Cottrell clouds. Every time a group of hydrogen-loaded dislocations during its migration reaches a cavity created by plastic deformation, a certain amount of hydrogen is discharged into the cavity. The increased pressure causes local triaxial stresses at the cavity and lowers the U.T.S. for the test bar as a whole. At low temperatures and high deformation speeds hydrogen cannot diffuse along with migrating dislocations, and no hydrogen embrittlement is caused. The way in which local stresses initiate fracturing is not explained.

PRESENT THEORY

A characteristic feature of hydrogen embrittlement is the lowering of the U.T.S.; fracture may occur either by shear or cleavage. Certainly both shear strength and cleavage strength are lowered by hydrogen. Unfortunately, the mechanism to initiate fracturing in steel is not satisfactorily understood, especially when fracture occurs by shear. The brittle fracture, however, is fairly well explained by the Griffith theory of fracturing. According to the original theory[16] the virgin material contains numerous small cracks. Fracturing will start at an external stress at which an incremental enlargening of a crack can result in a decrease of the total energy. This involves release of elastic strain energy in excess of the energy required to build up the new crack surface, so that the net energy set free in the incremental process is positive. When the crack can continue to spread at constant external stress with further decrease in total energy of the sample, sudden fracturing will occur at a very high rate of crack propagation.

The original theory was later modified by Orowan,[17] who showed that a certain plastic deformation will occur at the crack surface. Thus energy has to be supplied not only to create a new surface, but also for the plastic deformation. Hall[18] has calculated the amount of plastic work as a function of the rate of crack spreading.

It has never been fully understood how steel could contain original cracks and how these are related to the structure. Petch suggested an explanation in one of his recent papers.[19] The fracturing conditions are given by an array of dislocations which are blocked by a grain boundary, and therefore cause a stress concentration. This may either result in a slip in the new grain, or in the opening of a crack by cleavage. As Griffith's formula deals only with energy changes during crack spreading, the further spreading of a crack already initiated can then be interpreted in terms of the original Griffith theory.

If the material contains hydrogen, a crack, initially present or opened under stress or by deformation, will under equilibrium conditions be filled with hydrogen gas, the pressure of which is determined by the hydrogen content dissolved in the lattice. Part of the total energy of the test piece consists of the energy content of the gas in the cracks. On crack propagation the gas will expand with a corresponding release of energy. The released gas energy is added to the released strain energy; hence, the latter may now be smaller and still satisfy conditions for crack propagation. The amount of strain energy released increases with external stress. Hydrogen will thus lower the external stress at fracture.

This model requires the existence of a true crack, such as a Griffith crack, in which pressure can be built up. It is, however, not in disagreement with the dislocation array model even if no actual pressure can be built up in the dislocation array itself. Because of energy fluctuations there is always a tendency for a crack to open at the tip of the dislocation array, although at low stresses the crack will again disappear. If hydrogen can diffuse into an unstable crack, the energy conditions will become changed and the crack will be stabilized at a stress level that depends upon the hydrogen pressure, *i.e.*, the hydrogen activity of the lattice. The same reasoning can be applied to shear fracture. Whatever the conditions for the initiation of a crack by shear, the crack spreading has to follow the above-mentioned energy conditions. Cracks opened at low stresses are unstable if they cannot propagate, and will disappear. Hydrogen can, however, diffuse into such an unstable crack and stabilize it, which results in fracturing at a lower external stress.

The energy release, which has to be supplied by the expanding gas, may decrease to a certain extent with increasing crack size. With a very high rate of crack spreading, the gas can only expand adiabatically, which gives a particular falling curve for the released expansion energy as a function of the crack volume. Only for very low initial hydrogen pressures is the slope of this adiabatic curve less than the highest slope still capable of supporting crack propagation. The lowering of fracture stress can under these conditions be only slight. To obtain a greater lowering of the fracture stress, fresh hydrogen has to diffuse into the crack simultaneously with crack propagation, if the spreading of the crack is to continue. The lowering of the external fracture stress will still be determined by the initial gas pressure, but a certain amount of gas has to diffuse into the crack. The process of fracturing can thus be divided into two successive periods. During the first, the crack spreads slowly, the rate being determined by the rate of hydrogen diffusion. When the crack has grown sufficiently in size by this process the second period can begin : the crack then propagates with adiabatic gas expansion at a very high rate.

When the external stress increases instead of remaining constant during the first period, the second period will begin and fracture will occur at a higher external stress than that at which crack spreading started. This gives an explanation of why hydrogen embrittlement is less under impact tests or under tensile tests at low temperatures. The temperature of testing has yet another effect. If there is plastic deformation at the surface of the crack this will be a decreasing function of the rate of crack propagation. When temperature is lowered the hydrogen diffusivity decreases, resulting in a decreased rate of crack propagation during the first period. The total amount of work which must be done by external stresses and gas expansion will thus increase. Less hydrogen embrittlement will be observed at low temperatures, even at very low deformation speeds.

EXAMPLE FOR CALCULATIONS

The deduction of the present theory is somewhat different for different basic assumptions about initiation of fracturing. An example will be given here, the calculations for which are based on the assumption that a true Griffith crack already exists at the fracture stress, and is filled with hydrogen of equilibrium pressure. If according to the dislocation array model it is assumed that a stable or stabilized crack only forms at the fracture stress—which also may be true for shear fracture—only the relation between crack radius and volume will differ. This leads to somewhat different equations which, however, can be deduced in a similar way, as in the following example.

The material in question is assumed to contain a dish-like crack of radius r perpendicular to the external stress and filled with hydrogen gas. When the radius of the crack is increased by dr, the potential surface energy will be increased by

$$\frac{d}{dr}(T2\pi r^2)dr$$

where T is the surface energy per unit area.

During crack spreading the material will be deformed plastically near the surface of the crack,[17] the potential energy of the material being increased by

$$\frac{d}{dr}(\omega 2\pi r^2)dr.$$

On the other hand, the strain energy will be reduced on both sides of the crack to a distance comparable with the radius of the crack. The decrease in strain energy will be approximately equal to the original strain energy density multiplied by the volume of a

sphere of radius r, or

$$\frac{d}{dr}\left(-C\frac{4\pi r^3}{3}\cdot\frac{\sigma^2}{E}\right)dr$$

where C is a dimensionless constant of the order of unity.[20]

The potential energy of the gas decreases during expansion and causes an energy change of

$$-p\frac{dV}{dr}dr = -p\frac{d}{dr}(f\pi r^2)dr$$

where f is a dimensionless shape coefficient. The right-hand side of this equation is based on the assumption by Griffith that the relation between the length and thickness of the crack will not change during its propagation.

The condition for crack spreading is that the total energy change shall be negative for a positive value of dr:

$$\frac{d}{dr}\left(T2\pi r^2+\omega 2\pi r^2-C\frac{4\pi r^3}{3}\frac{\sigma^2}{E}-pf\pi r^3\right)\leqslant 0,$$

i.e.,

$$\frac{d}{dr}\left[(T+\omega)2\pi r^2-\left(\frac{\sigma^2}{E}+\frac{pf}{\frac{4}{3}C}\right)\frac{4}{3}\pi Cr^3\right]\leqslant 0.$$

Thus $(\sigma^2/E+3pf/4C)$ replaces σ^2/E of the original Griffith-Orowan formula.

If r_0 is the original crack radius, the cleavage strength of the hydrogen-free material σ_k is given by making $p=0$:

$$\sigma_k=\sqrt{\frac{E(T+\omega)}{Cr_0}} \quad \text{.....(3)}$$

When the crack contains hydrogen gas, the cleavage strength at a certain hydrogen pressure p can be expressed by the formula:

$$\sigma=\sqrt{\left[\frac{E(T+\omega)}{Cr}-\frac{3fE}{4C}p\right]} \quad \text{.....(4)}$$

First the condition for crack enlargement at sonic velocities is examined. The gas has to expand adiabatically and

$$\frac{p}{p_0}=\left(\frac{V_0}{V}\right)^{\gamma}=\left(\frac{r_0}{r}\right)^{3\gamma}$$

where p_0 is the pressure at the moment when crack spreading starts. This expression substituted into equation (4) gives

$$\sigma=\sqrt{\left[\frac{E(T+\omega)}{Cr}-kp_0\left(\frac{r_0}{r}\right)^{3\gamma}\right]}$$

where $k=3fE/4C$.

The crack enlargement will start when the external stress reaches σ with $r=r_0$. A condition for further spreading is that the external stress should never become smaller than the actual value of σ. Thus, at a constant external stress equal to σ at the moment when crack spreading starts,

$$\frac{d\sigma}{dr}\leqslant 0.$$

This leads to the condition

$$kp<\frac{E(T+\omega)}{3\gamma Cr}\left(\frac{r_0}{r}\right)^{3\gamma}.$$

The gas pressure when crack spreading starts is

$$kp_0<\frac{E(T+\omega)}{3\gamma Cr_0}=\frac{\sigma_0^2}{3\gamma}.$$

The crack can only spread with sonic velocities if the gas pressure does not exceed the value given by this equation. The critical value σ_{01}, to which the brittle cohesive strength can be lowered under these conditions, can be calculated by making $r=r_0$:

$$\sigma_{01}=\sqrt{\left[\frac{E(T+\omega)}{Cr_0}-\frac{E(T+\omega)}{3\gamma Cr_0}\right]}=\sigma_k\cdot\sqrt{\left(1-\frac{1}{3\gamma}\right)}=0{\cdot}87\sigma_k$$

Hydrogen can thus lower the cleavage strength under these conditions to 0·87 of its original value. The numerical value of σ_{01} depends on the original assumption on fracturing mechanism, and will be higher if the calculations are based on the dislocation array model.

At higher gas pressures and lower external stresses than those belonging to the critical value, crack spreading with adiabatic gas expansion cannot occur. To make crack spreading possible, gas diffusion to the crack, or heat conduction from the metal to the gas, is necessary.

The gas expansion in each interval dr is supposed to follow the polytropic equation $pV^n=$ constant. The polytropic exponent could have values between 0 and γ. The former corresponds to isobaric gas expansion, where the pressure of hydrogen is maintained constant by hydrogen diffusion from the lattice. It will, however, be shown later (*see* equation (13)), that the lower limit of the exponent must be set higher.

The crack condition may be written as

$$\sigma=\sqrt{\left[\frac{E(T+\omega)}{Cr}-kp_t\left(\frac{r_t}{r}\right)^{3n}\right]}$$

where r_t is an arbitrary crack radius greater than r_0, for which the conditions for further crack enlargement have been dealt with, and p_t is the corresponding gas pressure.

The condition for crack enlargement is as above, *i.e.*, $d\sigma/dr\leqslant 0$, which for $r=r_t$ leads to

$$n<\frac{E(T+\omega)}{3Cr_tkp_t}.$$

In this case, however, the sign $\leqslant$ is not valid. A smaller value of n than the highest possible would mean that the amount of hydrogen introduced in the crack by diffusion is higher than the amount necessary for crack spreading. Before this state could be achieved, however, the limit state, characterized by the sign $=$, must have been passed. At this point crack spreading actually occurs. The correct equation is therefore

$$n=\frac{E(T+\omega)}{3Cr_tkp_t}.$$

To find the relation between r, n, and p, the derivative of this equation for $r_t=r$ is combined with the derivative of the polytropic expansion energy equation

$$\frac{dn}{n}=\frac{d\omega}{T+\omega}-\frac{dr}{r}-\frac{dp}{p} \quad \text{.....(5)}$$

and

$$-\frac{dp}{p}=3n\frac{dr}{r} \quad \text{.....(6)}$$

The reason why ω has to be considered as a variable is that plastic deformation work, according to Hall,[18] is a decreasing function of the rate at which the crack

is spreading. However, the rate is in this case controlled by hydrogen diffusion and is much less than in the case of adiabatic gas expansion, leading to a higher value of ω. The exact value of ω and its variation during cracking cannot at present be calculated. In the following treatment it will be assumed that ω is constant, which makes the treatment strictly valid only for brittle fracture with $\omega = 0$. From an approximate calculation, however, important qualitative conclusions can be made on the expected deviations from the following simplified treatment.

The differential equations (5) and (6) lead to

$$C_1 r = \frac{3n-1}{n} \text{ and } C_2 p = \frac{1}{1-3n}.$$

The integration constants are determined in the starting moment by

$$n_0 = \frac{E(T+\omega)}{3Cr_0 kp_0} \quad \ldots\ldots(7)$$

After substituting in equation (3),

$$r = r_0\left(\frac{\sigma_k}{\sigma_0}\right)^2 \cdot \frac{3n-1}{3n} \quad \ldots\ldots(8)$$

and

$$kp = \sigma_0^2 \cdot \frac{1}{3n-1} \quad \ldots\ldots(9)$$

By eliminating n, the relation between p and r can be calculated:

$$kp = \sigma_k^2 \cdot \frac{r_0}{r} - \sigma_0^2 \quad \ldots\ldots(10)$$

From equation (8) it can be seen that n increases during the spreading of the crack, which means that hydrogen diffusion to the crack, necessary to maintain crack spreading, decreases. At a certain crack radius, $n = \gamma$ is reached, the crack can continue by spreading with adiabatic gas expansion, and hydrogen diffusion to the crack ceases. The diffusion-controlled crack spreading transforms to sudden fracture. The radius and pressure at this transformation are given by substituting $n = \gamma$ in equations (8) and (9):

$$r = 0{\cdot}76 r_0\left(\frac{\sigma_k}{\sigma_0}\right)^2 \quad \ldots\ldots(11)$$

and

$$kp = 0{\cdot}31\sigma_0^2 \quad \ldots\ldots(12)$$

Phragmén[13] calculated on a thermodynamic basis that hydrogen pressure can never exceed 48,800 atm. If this condition is satisfied for $\sigma_0 = 0$, fracture without external stresses can occur at sufficiently high gas pressures. The crack radius at transformation from diffusion-controlled spreading to sudden fracture is obtained by inserting $\sigma_0 = 0$ in equation (11), which gives $r = \infty$. The whole fracturing is thus diffusion-controlled. In this case n at the moment when crack spreading starts is given by

$$n_0 = \frac{E(T+\omega)}{3Cr_0 kp_0} = \frac{\sigma_k^2}{3kp_0} = \tfrac{1}{3}.$$

It can be shown that the same lower limit is obtained, even if variations of ω are taken into account. The limits for n given by

$$\tfrac{1}{3} \leqslant n \leqslant \gamma \quad \ldots\ldots(13)$$

are thus generally valid. It can be seen that crack spreading with isobaric gas expansion will never occur.

To explain the influence of temperature and deformation rate, the following must be taken into consideration. At very low deformation rates the consequences of lowering temperature will be that (*a*) the equilibrium gas pressure, according to the equation of Phragmén, will increase at a given hydrogen content, and (*b*) the pressure necessary for lowering the fracture stress by a certain amount will increase, if the plastic deformation work increases with lowered rate of crack spreading.

The latter effect can be shown qualitatively by making a rough approximate calculation; n is a measure of the amount of hydrogen which for a given crack radius has to diffuse into the crack to make its further spreading possible. Thus ω, which is determined by the rate of crack spreading, can be expressed by a suitable function of n, approximately by

$$\omega = \frac{\text{constant}}{n^a}$$

where a is a positive constant, increasing with decreasing temperature. Neglecting the surface energy in comparison with the plastic deformation work, the fracturing condition can then be expressed by

$$\sigma_0 = \sqrt{\left[\sigma_k^2\left(\frac{\gamma}{n_0}\right)^a - kp_0\right]}$$

where

$$n_0 = \frac{E\omega}{3Cr_0 kp_0}\left(\frac{\gamma}{n_0}\right)^a = \left(\frac{\gamma^a \sigma_k^2}{3kp_0}\right)^{\frac{1}{1+a}}$$

The condition for crack spreading in the diffusion-controlled range is given by

$$(kp)^{\frac{1}{1+a}} = \left(\frac{p_0}{p}\right)^a \left[(3\gamma)^a \sigma_k^2 \cdot \frac{r_0}{r}\right]^{\frac{1}{1+a}} - \left[(3\gamma)^a \sigma_k^2\right]^{\frac{1}{1+a}} + (kp_0)^{\frac{1}{1+a}}$$

As the rate of crack spreading is only influenced by hydrogen diffusion in the diffusion-controlled range, the application of these equations has to be limited to this range. The equations are thus valid only if the calculated fracture stress is lower than σ_{01}. The following qualitative conclusions can be drawn from the equations.

At the same gas pressure the fracture stress is higher than the value obtained when the effect of the rate of crack spreading on the plastic work in the surroundings of the crack is neglected. At the same equilibrium hydrogen pressure the radius at which adiabatic gas expansion starts becomes smaller and

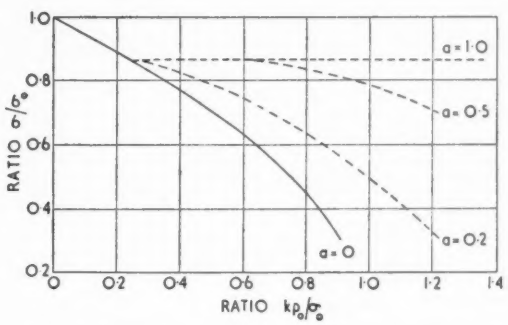

Fig. 1—Effect of hydrogen pressure on U.T.S.

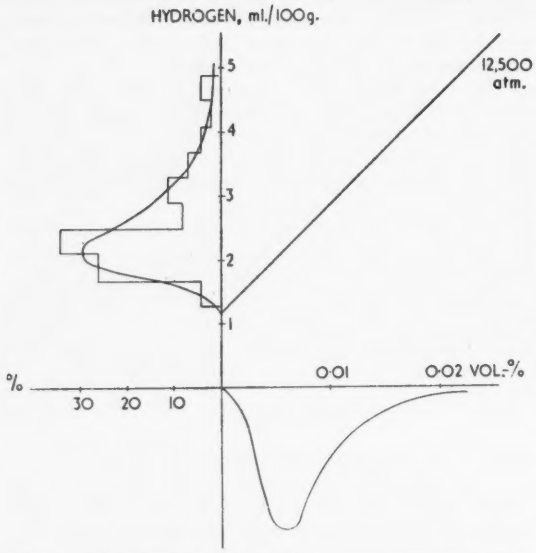

Fig. 2—Distribution of relative porosities in forged 1·75% nickel steel

the pressure higher than calculated previously, whereas the reverse is true for the same fracture stress. Generally, when the crack spreads from an initial radius and pressure, the pressure at a certain radius will be higher than that calculated on the simplified basis. The influence of the correction on fracturing conditions is shown in Fig. 1.

At high deformation rates or in normal tensile tests at low temperatures yet another effect must be taken into account. If $p > p_{01}$ the crack spreads in the first period at a rate controlled by diffusion. If the external stress increases simultaneously, the radius for transformation to sudden fracture will be reached at a higher external stress than the one corresponding to the hydrogen pressure at which crack spreading actually started. At a very high deformation speed the external stress can increase at fracture up to σ_{01}.

APPLICATION TO TENSILE TEST DATA

The model given to explain hydrogen embrittlement predicts that hydrogen will lower cleavage strength and shear strength. The intersection of the cleavage-strength/strain or the shear-strength/strain curve with the true-stress/strain diagram gives the U.T.S. This determines the reduction in area and elongation at fracture.

Though the true-stress/strain diagram is well known for many materials, very little is known about the cleavage-strength and shear-strength curves, except for very low temperatures. Assuming, however, that these are approximately straight lines in the significant part of the diagram, the calculated lowering of strength by hydrogen will be proportional to the lowering of the U.T.S. The lower the strain hardening of the material, the more a lowering of the U.T.S. will decrease ductility. The very high strain hardening of austenitic steels may thus explain their low susceptibility to hydrogen embrittlement.

To calculate the hydrogen pressure the amount of hydrogen dissolved in the lattice must be known. The amount not dissolved, included in cavities or trapped in or around dislocations, has to be subtracted from the total hydrogen content. For this purpose, as previously mentioned, all undissolved gas can be treated as if it were included in porosities. The relative volume of these porosities can be estimated by a statistical treatment of a large number of analyses in the same material. The distribution of porosities is not homogeneous, their relative volume varying somewhat in small limited volumes such as a sample for analysis. If equilibrium pressure is the same in the whole material the hydrogen content of the samples will follow variations in the relative volume of the porosities. The zero point of the frequency curve of hydrogen analysis distribution gives the hydrogen content dissolved in the lattice (*see* Fig. 2). From this the equilibrium pressure can be calculated. By projecting the frequency curve of analysis distribution over the line for constant pressure in the hydrogen-

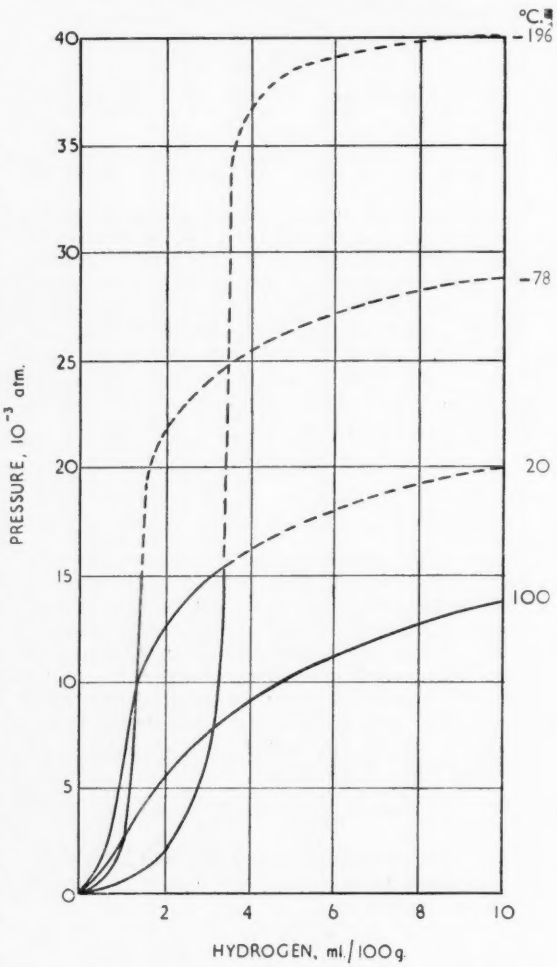

Fig. 3—Hydrogen pressure at 20° C. and 0·005 vol.-% porosities

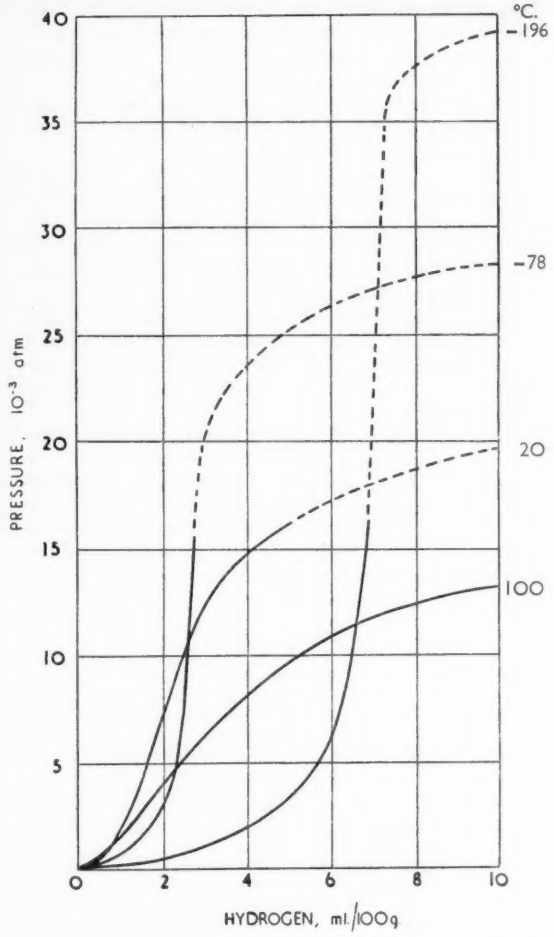

Fig. 4—Hydrogen pressure at 20° C. and 0·01 vol.-% porosities

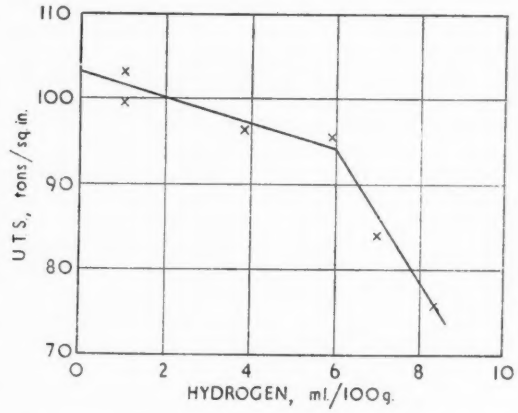

Fig. 5—Effect of hydrogen on the U.T.S. of a 3% Cr–Mo steel at — 196° C.

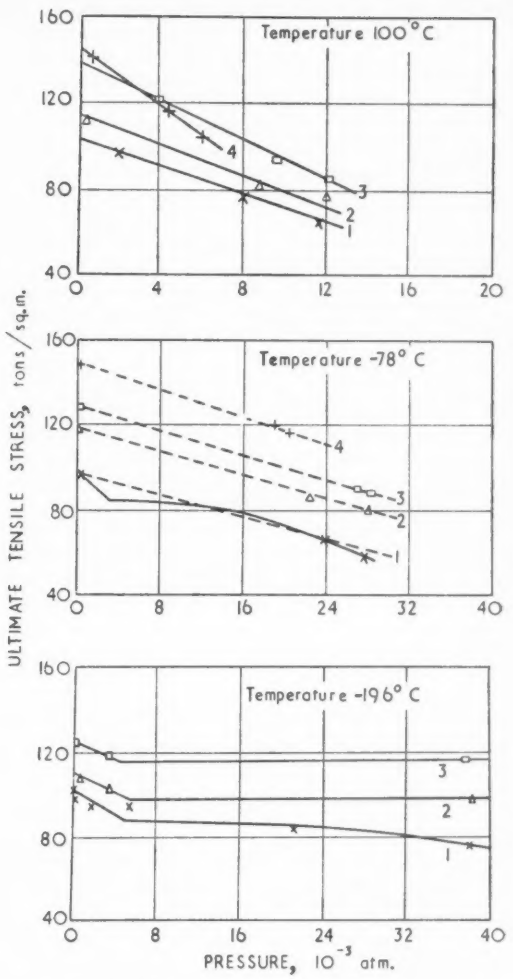

Fig. 6—Effect of hydrogen pressure, assuming 0·01 vol.-% porosity, on the U.T.S. of 3% Cr–Mo steel: (1) 41·5, (2) 53, (3) 67, and (4) 98 tons/sq. in.

content/porosity diagram, the frequency curve of porosity distribution can be determined.

Such a treatment has been carried out for the distribution of hydrogen analyses of specimens situated along the axis of large forgings.[21] It is shown for 47 specimens of a large 1·75% nickel steel forging in Fig. 2. The main value of porosity was 0·006 vol.-%; 80% of the specimens had a porosity between 0·0025 and 0·013 vol.-%. For partially forged ingots the main value was between 0·0035 and 0·0065 vol.-%, and for unforged ingots it was between 0·0085 and 0·018 vol.-%, but with a larger scatter in the values.

Though the scatter may be less for larger specimens, such as those used for tensile tests, it is a serious source of uncertainty when evaluating experimental results. The lower the testing temperature is, the greater will be the influence of porosity on the relation between hydrogen content and equilibrium pressure.

This relation is shown for 0·005 vol.-% in Fig. 3, and for 0·01 vol.-% in Fig. 4. Already at room temperature there is a significant difference up to 4 ml./100 g. By quantitative evaluations of experimental measurements, data may be used only in a range in which variations of porosity within the expected limits only slightly influence the equilibrium pressure. Thus, if low-temperature measurements are to be considered at all for such calculations, the hydrogen content must be correspondingly high.

The correction for rate-dependence of plastic deformation work at fracture has also to be taken into account. The correction is higher at low temperatures and is only known by its qualitative character. This is also an argument for using only measurements made at elevated temperatures. At these the simplified calculations are a good approximation.

Hobson and Hewitt[12] have examined the influence of hydrogen content on the strength and deformation properties in tensile tests for four different temperatures between — 196° C. and 100° C. Additional information about the U.T.S. and elongations at maximum stress have been made available,* from which the true-stress/strain diagrams were determined. First it could be corroborated that the diagrams were not altered by the hydrogen content at any of the temperatures investigated. Deviations were small and irregular and were probably caused by variations of properties of the different tensile bars. To facilitate further treatments, the experimental values of U.T.S. of hydrogenized bars were smoothed to make them fall on the true-stress/strain curve at the determined value of reduction in area.

Data at —196°C. reproduced in Fig. 5 for 41·5 tons/sq. in. (taken from Table V of the same paper[12]) show that porosity for this steel is about 0·01 vol.-%, as the sudden lowering of U.T.S. in the range 6–8 ml./100 g. can only be explained by a sudden increase of hydrogen pressure (*see* Fig. 4).

The relation between hydrogen pressure and U.T.S. at 100° C. (in water) at this value of porosity is shown for four tempered structures in Fig. 6. The heat-treatment of the samples was described by Hobson and Sykes.[3]

The curves at — 78° and — 196° C. are also shown, but care has to be taken in their evaluation for the above-mentioned reasons. It is, however, obvious that the correction for plastic deformation work becomes of greater importance at lower temperatures and perhaps also with harder structures. At — 196° C. and with the hardest structures the limiting value for decrease in U.T.S. seems to be σ_{01}.

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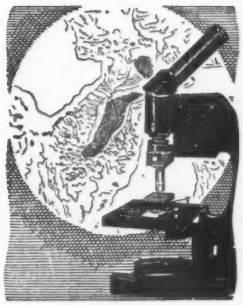

Tensile and Impact Properties of Fe–Si, Fe–Ni, Fe–Cr, and Fe–Mo Alloys of High Purity

By W. P. Rees, M.Sc., F.I.M., B. E. Hopkins, M.Sc., and H. R. Tipler, B.Met.

SYNOPSIS

The tensile and impact properties of normalized high-purity iron–silicon, iron–nickel, iron–chromium, and iron–molybdenum alloys at various temperatures covering the tough-to-brittle transition are discussed. The low brittle-fracture strengths and the high transition temperatures in the iron–silicon alloys are attributed largely to intergranular brittleness ; the large increase in transition temperature in tension and impact caused by increase in silicon content is presumably due mainly to the large rise in yield stress, since there was little change in the brittle strength. Refining the grain size of a 1% silicon–iron alloy eliminated failure at grain boundaries and resulted in a considerable fall in transition temperature compared with that of iron. The effect of nickel on high-purity iron in the brittle range is shown to be affected by two factors : the occurrence of grain-boundary weakness in alloys containing 2% of nickel and above, and the formation to some extent of α_2 structure. The brittle behaviour of iron–nickel alloys is sensitive to small amounts of certain elements such as carbon and oxygen, and the influence of nickel in accentuating the embrittling effect of oxygen is suggested as a cause for the occurrence of intergranular weakness in alloys with 2% of nickel and above. Evidence is given to show that the brittle strength of the iron–nickel alloys probably decreases with rise in temperature. Chromium and molybdenum were found to have only a small effect in reducing the impact transition temperature of high-purity iron.

A comparison is made of the influence of silicon, nickel, chromium, molybdenum, and manganese on the strength of high-purity iron in the ductile condition, and the strengthening effect of these elements for given weight percentages increases in the order chromium, molybdenum, nickel, manganese, silicon. A number of factors that may be involved in the strengthening produced by elements in solid solution are discussed, and it is suggested that a more precise knowledge of the particular type of lattice distortion involved is required before the factors responsible for the strengthening effect can be elucidated. It is emphasized that the softening effect of the first additions of chromium to iron now seems to be well established ; such effects need accounting for in any theory of the strengths of solid solutions.

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As part of a general programme on the influence of alloying elements on the mechanical properties of ferrite, the effects of a number of elements on the tensile and notched-bar impact properties of high-purity iron over a range of temperature spanning the tough-to-brittle transition have been studied, and the results on manganese, carbon, carbon + manganese, and oxygen have already been published.[1–3] The present paper deals with the results of a similar investigation on the effects of additions of up to 5% of silicon, nickel, chromium, and molybdenum on the properties of iron.

The work has been done on alloys prepared under conditions designed to give high purity on a scale large enough to enable extensive mechanical testing to be carried out. Special care has been taken to keep the level of impurities low and uniform in any one series of alloys. In general, impurities have been present in amounts smaller than 0·01%, but certain impurities have been kept much below this level.

PREPARATION OF ALLOYS AND TESTING PROCEDURE

The alloys were made from a special Swedish-iron base by the method described by Hopkins, Jenkins, and Stone,[4] consisting of first melting in air and then under an atmosphere of hydrogen in a 25-lb. H.F. induction furnace. The required percentage of alloying element was added after deoxidation of the iron, and melting was continued for a period to remove the

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Table I

CHEMICAL ANALYSES AND GRAIN SIZES OF IRON–SILICON ALLOYS

Element, %	34*AF*1 (1% Si)	33*AF*3 (2% Si)	33*AF*2 (3% Si)	33*AF*1 (4% Si)	32*AF*3 (5% Si)
Carbon	0·0016	0·0014	0·0026	0·0021	0·0024
Silicon	1·04	2·12	3·05	4·13	4·98
Manganese	←		<0·005		→
Sulphur	0·0052	0·0049	0·0049	0·0045	0·006
Phosphorus	n.d.	n.d.	n.d.	0·002	n.d.
Nickel	n.d.	n.d.	n.d.	0·005	n.d.
Chromium	n.d.	n.d.	n.d.	0·001	n.d.
Copper	n.d.	n.d.	n.d.	0·004	n.d.
Aluminium	0·001	0·002	0·001	0·001	0·004
Oxygen	0·0011	0·0009	0·001$_0$	0·0008$_7$	0·0011
Nitrogen	0·0013	0·0012	0·0014	0·0012	0·0006
Hydrogen	←		<0·000005		→
A.S.T.M. grain size	−1	−1	0	0	−1

n.d. = not determined

carbon and oxygen present in the addition. The metal was then cast under a small pressure of hydrogen into an octagonal steel mould $2\frac{3}{4}$ in. across the flats internally and $10\frac{1}{2}$ in. long without the feeder head.

The silicon* contained the following impurities:

C, %	Mn, %	P, %	Fe, %	Ni, %
0·02	0·001	0·009	0·009	<0·001
Cr, %	**Ti, %**	**Ca, %**	**Mg, %**	**Al, %**
<0·001	0·002	0·007	<0·001	0·02
	O_2, %	N_2, %	H_2, %	
	0·25	<0·01	0·003	

The nickel powder used* contained the following main impurities: C, 0·096%; Fe, 0·001%; Mo, approx. 0·001%; Si, 0·004%; O_2, 0·1%; Al, Cu, Mn, and S were not detected on 10-g. samples.

Electrolytic chromium, in the form of granules,* contained the following impurities: C, 0·005%; S, not detected on 10-g. samples; Si, 0·004%; O_2, 0·5%; and Fe, $<0·01\%$; spectroscopic analysis showed traces of Mn and Pb, the presence of Cu was doubtful, and other elements were not detected.

The molybdenum* was shown spectrographically to contain $<0·01\%$ of Fe, with traces of Ba, Cu, and Mg, whereas Ni, Si, Mn, Cr, Pb, and Al were not detected and the presence of vanadium was doubtful. Microchemical analysis of a small sample showed the phosphorus content to be $\gtrsim 0·001\%$ and, after pickling the sample in dilute HNO_3 to remove the surface, the carbon content was $\gtrsim 0·005\%$. Sulphur was not detected on a 10-g. sample, and other metallic impurities were said to be less than 0·002%.

The purity of the additions did not need to be quite as high as that of the iron, because only up to 5% of an alloying element was added. Nevertheless certain impurities in the additions, particularly oxygen and carbon, were considerably reduced in concentration by the method of preparation of the alloys, so that the final analyses of the alloys compared favourably with that of the high-purity iron base.

* The silicon was obtained from St. Lawrence Alloys and Metals, Ltd., Quebec, the nickel from the Mond Nickel Co., Ltd., the chromium from Johnson Matthey and Co., Ltd., and the molybdenum from the Tungsten Manufacturing Co.

The 25-lb. ingots of the alloys produced as described were hot-rolled to $\frac{5}{8}$-in. dia. bar and normalized by heating at 950° C. for a short time and allowing to cool freely in air.

Tensile tests on all alloys were carried out in a hydraulic testing machine over a range of temperature from room temperature to − 196° C. in the apparatus, and using the coolants described in a previous paper.[1] Experiments with the iron–silicon alloys required tests to be done above room temperature, and for this purpose an oil bath electrically heated and continuously stirred was used to attain temperatures up to 200° C. Proof stresses were determined at all temperatures using a specially designed extensometer,[3] except in the case of certain iron–nickel alloys when, at room temperature and − 73° C., the minimum diameter of the test piece was continuously measured simultaneously with the load, enabling true-stress/natural-strain curves to be drawn. The tensile test pieces machined from the normalized bars had a gauge dia. of 0·282 in., and a gauge length of 1 in., with $\frac{1}{2}$-in. B.S.W. threads.

A Charpy-type machine was used for a series of impact tests on each alloy at temperatures covering the tough-to-brittle fracture transition using Charpy test pieces 55 mm. long × 10 mm. square with an Izod **V** notch. In view of the gradual nature of the change from tough to brittle fracture in the iron–silicon and iron–nickel alloys, the transition temperature has been taken as that at which the impact value was half the value in the fully tough condition.

IRON–SILICON ALLOYS

The chemical analyses of alloys containing up to 5% of silicon are given in Table I. Phosphorus, nickel, chromium, and copper were not determined in all the alloys because the proportions of these elements in iron alloys made in the 25-lb. H.F. furnace used in this work are always approximately those given for the 4% silicon alloy. The impurities were at a uniform level in all alloys, and in particular the carbon, oxygen, and nitrogen contents were low. The A.S.T.M. grain sizes of the alloys in the normalized condition are given in Table I.

Tensile Tests

The results of the tensile tests are given in Figs. 1*a–d*. As a definite yield was not usually observed, the 0·5% proof stress of the five alloys has been plotted against temperature in Fig. 1*a*. At all temperatures at which a proof stress could be measured, *i.e.*, at temperatures at which a specimen was not completely brittle, the effect of silicon was to raise it markedly. Thus, at room temperature it rose from 11 tons/sq. in. for the 1% alloy to 33 tons/sq. in. for the 5% alloy. There was an increase in proof stress as the temperature was reduced, this effect being somewhat greater at the lower temperatures.

From Fig. 1*b* it will be seen that the U.T.S. of the 1% silicon alloy rose gradually as the temperature fell from 100° to — 196° C., that of the 2% alloy remained sensibly constant from 200° to — 196° C., and there was a fall in the strength of the 3%, 4%, and 5% alloys over this range of temperature, the rate of fall being greatest for the 5% alloy. Silicon increased the tensile strength over most of the temperature range; *e.g.*, the strength of the 5% alloy was nearly double that of the 1% alloy at 100° C., but at — 196° C. the strengths of all the alloys lay within the range 24–33 tons/sq. in.

The percentage elongation and reduction in area are plotted against temperature in Figs. 1*c* and *d*. The curves are similar in form to impact-value/temperature curves, in that there is a temperature below which there is a rapid fall in ductility. The rapid fall in elongation generally started at a lower temperature than that for reduction in area. The initial fall in ductility approximately coincided with the onset of brittle fracture, and in this temperature region test pieces broke in a brittle manner even when considerable necking had taken place. The temperature at which the ductility started to fall rapidly increased with rise in silicon content; using the data

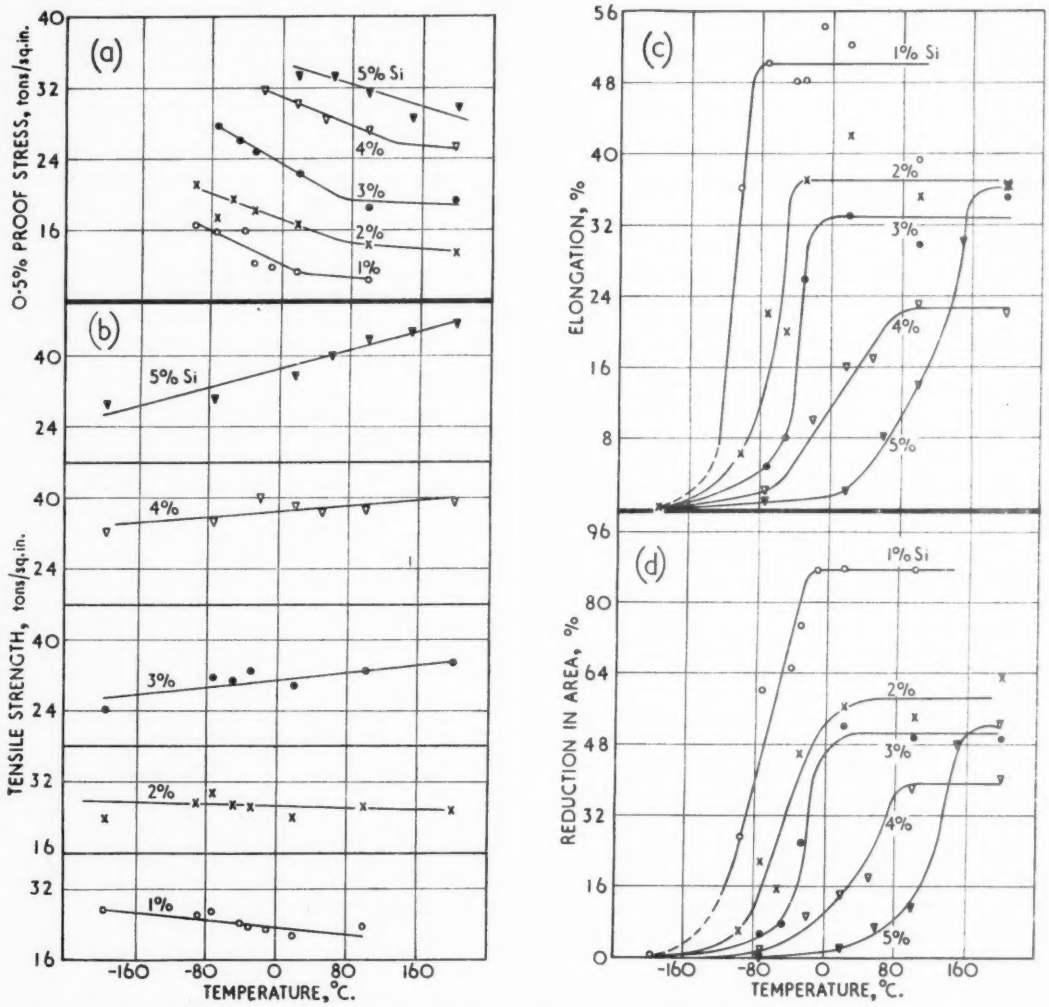

Fig. 1—Tensile properties at various temperatures of iron–silicon alloys normalized at 950° C.: (*a*) 0·5% proof stress; (*b*) tensile strength; (*c*) elongation; (*d*) reduction in area

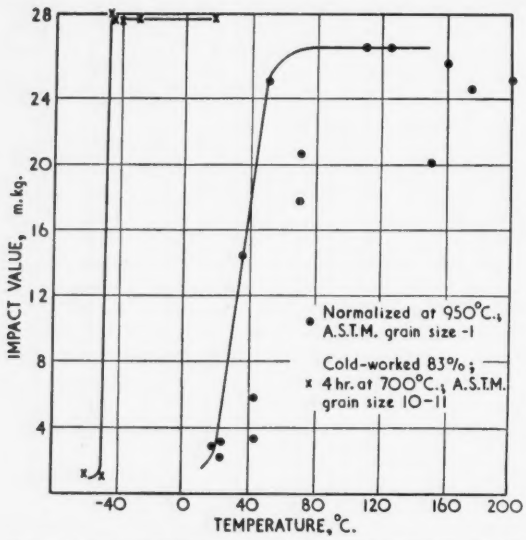

Fig. 2—Impact values at various temperatures of 1% silicon alloy of two grain sizes

for elongation, the actual temperatures were approximately — 73°, — 30°, 0°, 100°, and 200° C. for the 1–5% alloys, respectively.

The elongation and reduction in area obtained in the fully ductile condition decreased progressively with increase in silicon content up to 4%. The 5% alloy was, however, appreciably more ductile than the 4% alloy above about 130° C. At — 196° C., where fracture occurred without deformation in all the alloys, the brittle-strength value was the same as the tensile strength, and it did not appear to be very dependent on the silicon content. It has been shown[1] that the brittle strength of iron of high purity at — 196° C. is about 45 tons/sq. in. if fracture occurs entirely by cleavage, but if failure starts at some grain boundaries, although it continues partly by cleavage, the brittle strength may fall well below this value. Whereas intergranular fracture can be recognized readily in iron of moderate grain size, it is more difficult in iron–silicon alloys, presumably owing to differences in the shape of the grain-boundary surfaces. Examination of the fractures led to the conclusion that the 1%, 2%, and 5% alloys failed partly along the grain boundaries, although the 5% alloy was more difficult to assess than the other two. Areas in the fractures of the 3% and 4% alloys had characteristics which could not be attributed to cleavage and were almost certainly intergranular. The main characteristic was a curvature similar to that of areas which were definitely intergranular in the other alloys, but the usual glistening appearance was not well defined. This incidence of intergranular weakness is therefore considered to account for the low brittle strengths of all the iron–silicon alloys at — 196° C.

It is possible to deduce that the brittle strength of the alloys decreased with fall in temperature if the proof or yield stresses at a temperature just above that at which the alloys were completely brittle are compared with the strength at, say, — 196° C. Thus, at — 73° C. the proof stress of the 3% alloy was 27·6 tons/sq. in., so that the brittle strength at this temperature could not be less than this; at — 196° C. the strength was 24·4 tons/sq. in. For the 5% alloy the yield stress at 17° C. was 33–34 tons/sq. in., whilst the brittle strength was 30·3 tons/sq. in. at — 73° C. and 29 tons/sq. in. at — 196° C. The weight of this argument is somewhat affected by the difficulty of assessing the strength in the completely brittle condition because of scatter in the figures o' tained in this temperature region, most probably owing to intergranular weakness.

Charpy Impact Tests

The tough-to-brittle transition in impact was determined for each of the five alloys, a typical impact-value/temperature curve (that of the 1% alloy) being shown in Fig. 2. The form of the curve differs from that of high-purity iron[1] in the extended range over which the transition occurs. There was also a considerable scatter in the results in the whole temperature range over which the tests were carried out. Whereas in high-purity iron the sharp transition is marked by onset of fracture by cleavage only, the fractures of the iron–silicon alloys were in part intergranular, and this is thought to account for the transition covering a range of temperature. It has been pointed out[1] that for iron a combination of coarse grain size and fracture in part along grain boundaries gives less regular results in impact than are obtained in finer-grained material, and it is thought that these two factors, *i.e.*, large grain size and grain-boundary weakness, were responsible for the scatter in these alloys.

However, the results (Fig. 3) show that silicon causes a very rapid rise in the transition temperature in impact. The 2% silicon alloy had a somewhat higher transition temperature than would be expected from its silicon content, but it was nevertheless slightly lower than that of the 3% alloy. In this connection, Yensen[5] found that alloys containing about 2·6% of silicon were substantially more brittle than alloys containing less or more silicon.

Grain Refinement in Iron–Silicon Alloys

It has been shown[1] that intergranular brittleness can be made more pronounced by coarsening the grains; if the grain-boundary weakness is caused by

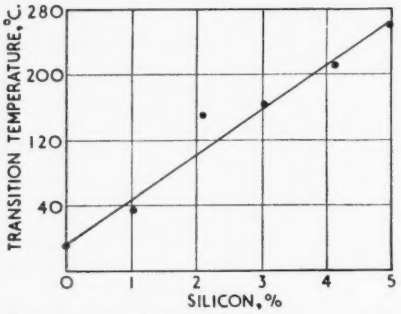

Fig. 3—Impact transition temperatures of iron–silicon alloys normalized at 950° C.

Table II

TENSILE AND IMPACT PROPERTIES OF 1% SILICON–IRON ALLOY OF TWO GRAIN SIZES

Property	Normalized at 950° C. (A.S.T.M. Grain Size −1)			83% Cold Work, 4 hr. at 700° C., Air-Cooled (A.S.T.M. Grain Size 10–11)		
	Room Temp.	−73° C.	−196° C.	Room Temp.	−73° C.	−196° C.
Upper yield, tons/sq. in.	11·1	15·8	...	18·2	24·8	43·8
Lower yield, tons/sq. in.	10·9	15·5	...	17·3	23	38·8
U.T.S., tons/sq. in.	21·6	27·0	27·3	24·4	29·2	42·6
Elongation on $4\sqrt{\text{area}}$, %	52	50	0	59	58	48
Reduction in area, %	87	60	0	92	87	64
Fracture stress at − 196° C., tons/sq. in.	...	...	27·3	...	...	89·6
Fracture	Ductile	Brittle + necking	Brittle	Ductile	Ductile; starred	Brittle + necking
Impact transition temperature, °C.	34			−49		

traces of impurities, a larger area of grain-boundary surface (*i.e.*, finer grains) would be expected to minimize the effect. An attempt was therefore made to produce fine grains, by cold-rolling and annealing, in each of the iron–silicon alloys, to try to eliminate intergranular weakness and thus determine the effect of silicon on cleavage strength as distinct from intergranular strength, which is the property mainly reflected by the results of the coarse-grained material tested in the brittle condition.

1% *Silicon–Iron Alloy*—Cold-rolling was started with the bars at 100° C., but it was found that reheating was unnecessary and rolling was satisfactorily completed at room temperature with a total reduction of 83%. A treatment of 4 hr. at 700° C. followed by air-cooling was adopted to give uniform fine grains of A.S.T.M. size 10–11 (103 grains/mm.).

2% *Silicon–Iron Alloy*—Attempts to roll this alloy at 150° C. were unsuccessful, and could not be pursued at higher temperatures owing to lack of material.

3%, 4%, *and* 5% *Silicon–Iron Alloys*—A 0·9-in. dia. bar of the 4% alloy made a creaking noise at the third pass when rolled at 250° C., and microscopic examination revealed numerous Neumann bands in some of the grains. A second bar behaved similarly. When rolling was continued at this temperature the bar cracked at the next pass. Similar cracking occurred during rolling at 350° C. Examination of the fractures obtained by breaking the bar through the rolling cracks showed that cracking started at the surface mainly along grain boundaries, but also to some extent along cleavage planes. Rolling at a still higher temperature of 600° C. of 1¼-in. dia. bars of the 3%, 4%, and 5% silicon alloys also proved unsuccessful. In view of the possibility that the hot work that had been done on the 0·9-in. and 1¼-in. dia. bars was insufficient to enable them to be satisfactorily 'cold-rolled,' further experiments were done on material that had been previously hot-rolled to ⅝ in. dia.

Lengths of ⅝-in. dia. bar were heated to 750° C. and given approximately 10% reduction when surface cracks appeared. These were filed out but, after another two passes with intermediate reheating, deeper cracks formed. At 800° C. by reheating after going twice through each pass (5% reduction) and filing out surface cracks as soon as they appeared, the bars were reduced 50%. One half of a bar of each alloy was thoroughly cleaned and rolling continued by the same procedure. No further cracks formed and a total reduction of 75% was obtained. Microscopic examination showed that a certain amount of recrystallization had occurred during rolling at 800° C., and, in an attempt to recrystallize completely and refine the grain structure, heat-treatments for 1 hr. at 700°, 750°, and 800° C. were given to each alloy after 50% and 75% reduction. There was, however, little further recrystallization or grain refinement, except possibly for the alloys given 75% reduction and annealed at 800° C. From these results it had to be concluded that the production of fine grain size in the 3%, 4%, and 5% silicon alloys by this 'cold-working' and annealing method held very little promise. Inability to roll below the recrystallization temperature without cracking precluded the application of sufficient severe deformation to produce fine grains on annealing. The occurrence of grain-boundary weakness in the iron–silicon alloys may have adversely influenced their ability to be cold-worked, the larger proportion of the fracture surface of the cracked bars being grain interfaces.

It is of interest that the 3% silicon alloy could be rolled cold with ease in sheet form, reductions up to 90% without intermediate annealings being obtained after hot-working at 1100° C. to strip about 0·013 in. thick. Initially the strip was heated to 100° C., but after a small amount of reduction reheating was found to be unnecessary.

To check that the 3% silicon alloy was not unusual in this respect, some sheet-rolling experiments were carried out on the 4% and 5% silicon alloys. It was found that, whereas these alloys could also be rolled at room temperature in sheet form, this was only possible after preliminary extensive reduction at 400–500° C. to a thickness of about 0·02 in.; in this connection, the 5% alloy needed more careful handling and a larger ultimate reduction before the material could be rolled at room temperature. In other words, there was a progressive increase with silicon content

Table III

CHEMICAL ANALYSES AND GRAIN SIZES OF IRON–NICKEL ALLOYS

Element, %	53*AF*1 (Iron)	79*AF*1 (1% Ni)	36*AF*1 (2% Ni)	36*AF*2 (3% Ni)	36*AF*3 (4% Ni)	37*AF*1 (5% Ni)	34*AF*3 (1% Ni + 0·01% O_2)	73*AF*2 (1% Ni + 0·005% C)	62*AF*1 (1% Ni + 0·013% C)
Carbon	0·0025	0·003	0·002	0·0013	0·0016	0·0022	0·0021	0·005	0·013
Silicon	0·003	0·009	0·004	0·004	0·003	0·005	0·006	0·006	0·003
Manganese	←				<0·005				→
Sulphur	0·0046	0·0049	0·004	0·0042	0·0046	0·0042	0·0046	0·006	0·0046
Phosphorus	0·001	n.d.	n.d.	n.d.	n.d.	n.d.	<0·001	n.d.	n.d.
Nickel	0·007	1·00	2·2	2·96	3·98	4·96	1·1	1·02	0·97
Chromium	0·001	n.d.	n.d.	n.d.	n.d.	n.d.	0·001	n.d.	n.d.
Copper	0·005	n.d.	n.d.	n.d.	n.d.	n.d.	0·004	n.d.	n.d.
Aluminium	0·004	0·0026	n.d.	n.d.	n.d.	<0·001	<0·001	<0·001	0·01
Oxygen	0·001	0·0019	0·0014	0·0014	0·0015	0·0014	0·01	0·0016	0·0006
Nitrogen	0·001	0·0009	0·0008$_6$	0·001	0·0008	0·001	0·0027	0·0019	0·0013
Hydrogen	←				<0·000005				→
A.S.T.M. grain size (normalized at 950° C.)	2–3	4–5	4	5	>6	>6	4	5	5–6

n.d. = not determined

in the temperature at which cold-rolling could be carried out; progressively larger reductions were also required before rolling was possible at room temperature. It appeared that in a certain thickness range during reduction the minimum temperature at which 'cold-rolling' could be carried out satisfactorily dropped fairly rapidly to room temperature. It is thought that the difficulty of cold-rolling the iron–silicon alloys at temperatures above that at which they first become fully ductile in the impact test is determined at least in part by the incidence of intergranular brittleness.

Mechanical Properties of Fine-Grained 1% Silicon Alloy

The results of tensile tests at room temperature, — 73°, and — 196° C. on the 1% silicon alloy in the fine-grained condition (A.S.T.M. grain size 10–11) produced by cold-rolling and annealing are given in Table II. Comparison with the tensile properties of the normalized 1% silicon alloy shows that grain refinement has resulted in an increase in tensile strength and a larger increase in yield stress at room temperature and — 73° C. The elongation and reduction in area were also somewhat higher in the fine-grained material at room temperature. At — 73° C. the fine-grained material was completely ductile with a 'star' fracture (*i.e.*, radial cracking), whereas the normalized alloy failed with a brittle fracture after necking. The effect of grain refinement in depressing the temperature of transition from tough to brittle fracture in tension was further shown by the results at — 196° C.; the normalized material broke in a completely brittle manner with a low tensile strength characteristic of partly intergranular failure and the fine-grained material broke with a completely cleavage fracture at a much higher stress after elongating and reducing in area to a large extent. The drop in transition temperature brought about by grain refinement was to a large extent the result of the elimination of the tendency to fail along grain boundaries, but it might also have been due in part to a small grain size *per se*. A comparison of the differences between the upper and lower yield points of the fine-grained alloy at room temperature, — 73°, and — 196° C., *viz.*, 0·9, 1·8, and 5·0 tons/sq. in., respectively, shows the effect of decreasing temperature in promoting a more pronounced yield.

As shown in Table II, grain refinement caused a reduction of over 80° C. in the transition temperature in impact. The fractures of the fine-grained material in the brittle condition occurred by cleavage as distinct from the partly intergranular failures obtained in the normalized condition. Conforming with this change in mode of fracture, the impact-value/temperature curve (*see* Fig. 2) gave a sharp transition from tough to brittle fracture in the fine-grained condition, whereas the transition was more gradual in the normalized condition.

It is of interest that the transition temperature of the fine-grained 1% silicon alloy was lower than that of high-purity iron in the normalized condition (A.S.T.M. grain size 2–3) in both tension and impact. This might well be due to difference in grain size. Experiments designed to produce iron having a grain size as fine as that of the 1% silicon alloy have not yet been successful, so that the extent of the assumed drop in transition temperature with decrease in grain size is not known.

Consideration of Results

The results show the very marked effect that silicon has in raising the temperature at which brittle failure will occur. The tensile tests carried out at — 196° C. demonstrated that increasing the silicon content from 1% to 5% certainly did not decrease the brittle strength at this temperature. The rise in transition temperature with silicon content in these alloys must therefore be attributed to the increase in proof stress. The addition of 1% of silicon to iron, in the normalized condition, resulted in an increase in proof stress which was insufficient to account for the large increase in transition temperature, and this was probably due to a greater extent to the decrease in brittle strength

from about 45 tons/sq. in. for an iron giving entirely cleavage fracture to 26 tons/sq. in. for the 1% silicon alloy, the fracture of which was partly intercrystalline.

Similar low brittle strengths associated with intergranular failures have been obtained previously[1, 2] in iron–oxygen alloys with oxygen contents above 0·003%. There is no direct evidence for the effect of oxygen in iron–silicon alloys, the oxygen contents being uniformly very low, but it is possible that some alloying elements as, for example, silicon, may decrease the oxygen content above which intergranular weakness is encountered. On the other hand, intergranular brittleness in the iron–silicon alloys might be due to silicon itself as distinct from any effect it might have on the distribution of traces of impurities. However, the elimination of intergranular brittleness brought about by decreasing the grain size of the 1% silicon alloy is consistent with the effect being due to traces of impurities. The consequent decrease in transition temperature might have been partly due to grain size as such. In this connection, however, it is of interest that Petch (*see* Allen[6]) found no effect on the brittle-fracture stress at liquid nitrogen temperature of grain-size variation produced by straining and annealing decarburized mild steel.

IRON–NICKEL ALLOYS

The chemical analyses of the alloys containing up to 5% of nickel are given in Table III, from which it will be seen that the impurities are uniformly low. Three 1% nickel alloys, 34*AF*3, 73*AF*2, and 62*AF*1, containing 0·01% of oxygen, and 0·005% and 0·013% of carbon, respectively, are included in the series.

Since it is known that the distorted α_2 structure is formed in iron–nickel alloys, tempering treatments for various times at 550°, 600°, and 650° C. were carried out on small specimens of the normalized material and hardness tests were made so as to give an indication of the removal of the distortion in the lattice. The hardnesses after these treatments are given in Fig. 4.

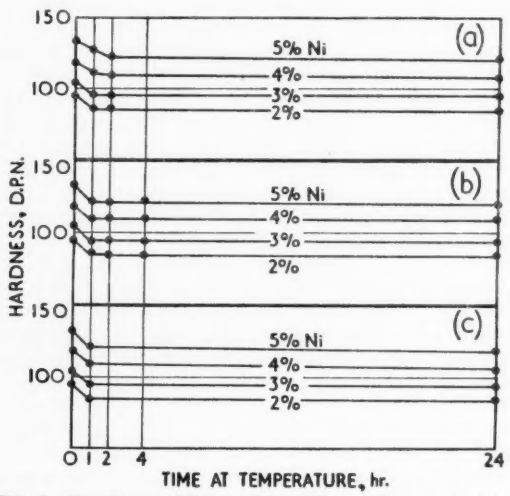

Fig. 4—Hardness of iron–nickel alloys tempered at (*a*) 550°, (*b*) 600°, and (*c*) 650° C.

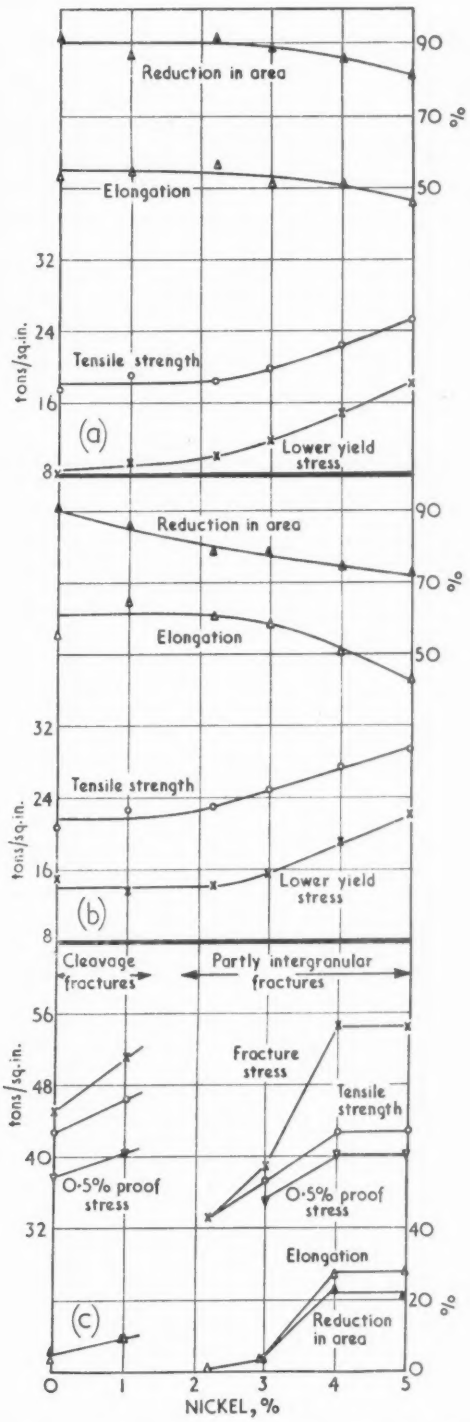

Fig. 5—Tensile properties at (*a*) room temperature, (*b*) — 73° C., and (*c*) — 196° C. of iron–nickel alloys normalized at 950° C.

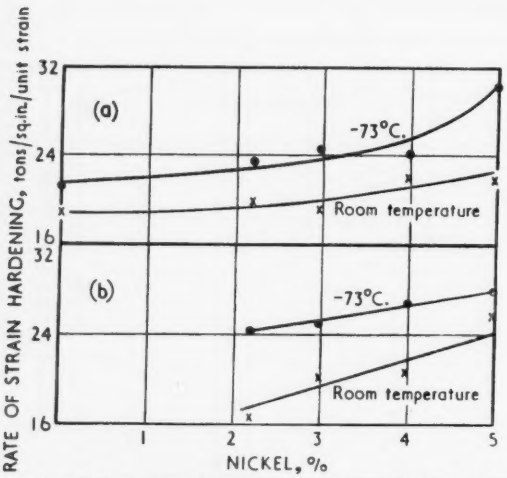

Fig. 6—Rate of strain hardening of iron–nickel alloys (*a*) normalized at 950° C., (*b*) tempered 4 hr. at 600° C., air-cooled

Unlike the iron–manganese alloys previously investigated,[1] there was only a small drop in hardness on tempering at any of these temperatures and the effect did not increase with nickel content. Nevertheless, the microstructures and mechanical properties of the normalized alloys suggested that the distorted α_2 structure was present in a sufficient degree to affect some of the results, so that tensile and Charpy impact tests were carried out on the alloys after tempering for 4 hr. at 600° C. and air-cooling as well as on the alloys in the normalized condition.

For comparison with alloys in the normalized condition, tensile and impact properties of a high-purity iron are included in the results given in this paper. The effects of the impurities oxygen and carbon on the properties of the 1% nickel alloys are also discussed.

Microstructures

The microstructures of the low-nickel alloys, of which that of the 2% nickel alloy shown in Fig. 10*a* is representative, consisted of polygonal grains of ferrite. With increasing nickel content, the grain size (*see* Table III) decreased and the grain boundaries became jagged. It was noted in previous work on iron–manganese alloys,[1] which were known to consist of α_2, that one of the characteristics of the structure was an irregularity of the grain boundaries, which was probably connected with the acicular nature of the α_2. A similar irregularity of grain boundaries was apparent in the higher-nickel alloys. Acicular markings inside the grains in the higher-nickel alloys could not be identified beyond doubt because there seemed also to be a small amount of coring (*see* Fig. 10*b*). The liquidus and solidus curves of the iron–nickel system do not suggest that there should be big differences in the extent of coring as the nickel content is increased from 1% to 5%. It was considered, therefore, that the characteristics of the α_2 structure found in the microstructures of the higher-nickel alloys, together with the effects noted in the mechanical properties of the alloys in the normalized condition, left little doubt of its presence.

Tensile Tests

The results of tensile tests at room temperature and — 73° C. on the material in the normalized condition are given in Figs. 5*a* and *b* and Fig. 6*a*. The effect at room temperature and — 73° C. of increasing the nickel content was to increase the yield stress and tensile strength and to decrease the ductility slightly. Lowering the temperature of test to — 73° C. resulted in an increase in the yield stress and tensile strength. Whereas the reduction in area had decreased with fall in temperature to — 73° C. for all the alloys, the elongation had increased for the 1%, 2%, and 3% nickel alloys, was the same for the 4% alloy, and was slightly less for the 5% alloy. All the fractures were ductile and of the cup-and-cone type, but with the appearance at — 73° C. of radial cracking or 'star' fractures in the 3–5% nickel alloys, a feature which became more pronounced with increasing nickel content. It is of interest that star fractures had earlier been obtained[1] at — 73° C. with the 3% and 5% manganese alloys after normalizing, in which condition they contained α_2 structure.

The rates of strain-hardening as determined from the slope of the linear portion of the true-stress/natural-strain curves at room temperature and — 73° C. are shown in Fig. 6*a*, from which it is seen that both decrease in testing temperature and increase in nickel content produced an increase in the rate of strain-hardening.

The results of the tensile tests at — 196° C. on the normalized alloys are given in Fig. 5*c*. The results fall into two classes as a direct consequence of a change in mode of brittle fracture at a nickel content of 1–2%. Up to 1% of nickel the fractures were entirely of the cleavage type consistent with fracture stresses of 45–51 tons/sq. in. The effect of adding nickel to iron in this range was to raise the 0·5% proof stress, tensile strength, fracture stress, and ductility at — 196° C. At nickel contents of 2% and above, fracture occurred to a considerable extent along grain boundaries in addition to cleavage planes; this was evident from the appearance of the fracture surfaces under a low-power binocular microscope in the 2% and 3% nickel alloys, but owing to increasing fineness of the grains it was necessary to resort to microscopic examination of sections through the fractures for the 4% and 5% nickel alloys (*see* Fig. 11). Initial fracture along grain boundaries in preference to cleavage planes indicated that the grain-boundary strength was less than the true cleavage strength, and the fracture stresses of the 2% and 3% nickel alloys were considerably lower than would be expected if fracture had started along cleavage planes, as in the iron and 1% nickel alloy. The ductility of the 2% and 3% nickel alloys was also very low. Further increase in nickel content gave considerably higher fracture stress, tensile strength, and ductility. Proof stresses were obtained at 3% of nickel and above, and they increased with nickel content; there was a suggestion of a yield in the 4% and 5% nickel alloys. The fracture stresses of 54–55 tons/sq. in. in the 4% and 5% nickel alloys were less than would have been expected if fracture had occurred by cleavage only, but they were well above the cleavage strengths of

the iron and 1% nickel alloy. It should be noted, however, that these are fracture stresses of material which had been work-hardened substantially during test. The improvement in ductility at — 196° C. as the nickel content was raised indicates that the transition temperature in tension was lowered for both types of fracture.

The results of tensile tests at room temperature and at — 73° C. on nickel alloys which had been tempered at 600° C. after normalizing are given in Figs. 7*a* and *b* and Fig. 6*b*. This treatment had little effect on the tensile properties at these temperatures. All the fractures were ductile and star fractures still occurred at — 73° C. in the 3–5% nickel alloys, the radial cracks being at least as marked as in the normalized alloys. The rate of strain-hardening was raised by increase in nickel content and decrease in temperature of test, as shown in Fig. 6*b*. The effect of tempering on the tensile properties at — 196° C. (Fig. 7*c*) was generally small. All the specimens broke with a brittle fracture with similar evidence of grain-boundary failure in the 2–5% alloys as in the normalized alloys.

Charpy Impact Tests

The tough-to-brittle transition in impact was determined for each of the alloys in the normalized and in the tempered conditions. The impact-value/temperature curves for the 1% and 3% nickel alloys in the normalized and tempered conditions are shown in Figs. 8*a* and *b*, the latter being typical of the 2–5% alloys. The 1% nickel alloy had a sharp transition from tough to brittle fracture, whereas with the 3% alloy the transition occurred over an extended range of temperature in both conditions, the gradual nature of this being somewhat more pronounced with the tempered alloy. Compared with the completely cleavage fractures of the 1% nickel alloy, all broken test pieces of the 2–5% alloys showed both intergranular and cleavage fracture and, as has been suggested in connection with the iron–silicon alloys, the presence of grain-boundary weakness is thought to account both for the scatter of the results of individual tests and for the form of the impact-value/temperature curves of the 2–5% nickel alloys.

The effect of increasing nickel content in these alloys in the normalized condition (Fig. 9) was to increase the transition temperature in impact, the most marked rise occurring as the nickel content rose from 1% to 2%, coinciding with the appearance of intergranular brittleness. This is the reverse of the trend of transition temperature in tension as revealed by tensile tests at — 196° C., in that the alloys having the maximum ductility in tension at this temperature had the highest transition temperature in impact.

The effect of tempering at 600° C. on the transition temperature in impact was complex, and different for the low-nickel alloys, which gave completely cleavage fractures in the brittle specimens, and for the higher-nickel alloys, in which brittle fracture started along grain boundaries. The high-purity iron was not tempered, but the results for the 1% nickel alloy showed that tempering had only a small effect on the transition temperature. The results for the 2–5% nickel alloys suggested that two opposing

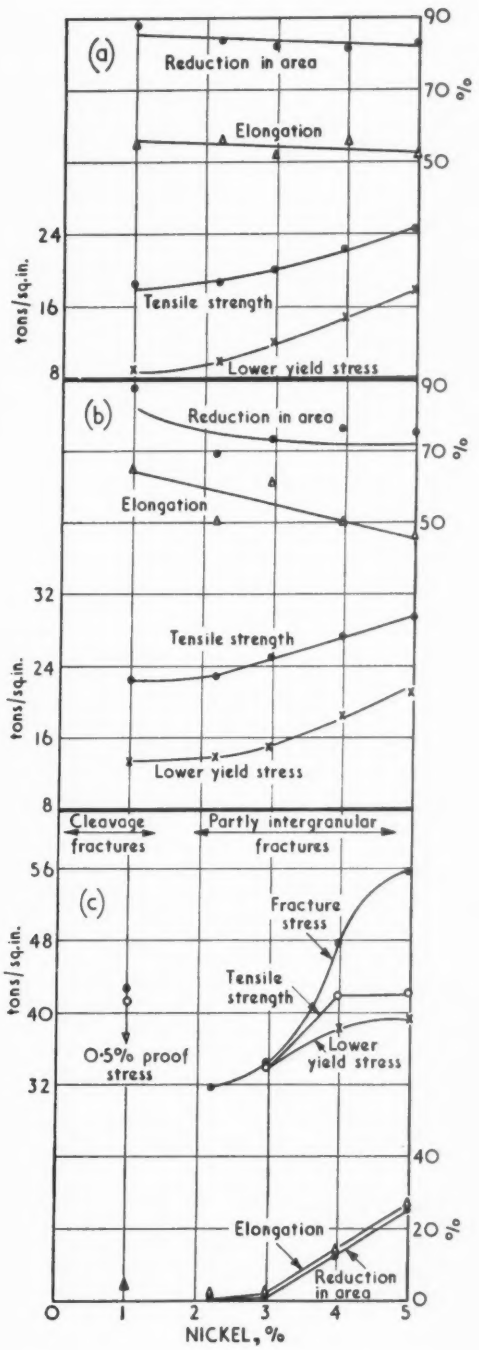

Fig. 7—Tensile properties at (*a*) room temperature, (*b*) — 73° C., and (*c*) — 196° C. of iron–nickel alloys tempered 4 hr. at 600° C., air-cooled

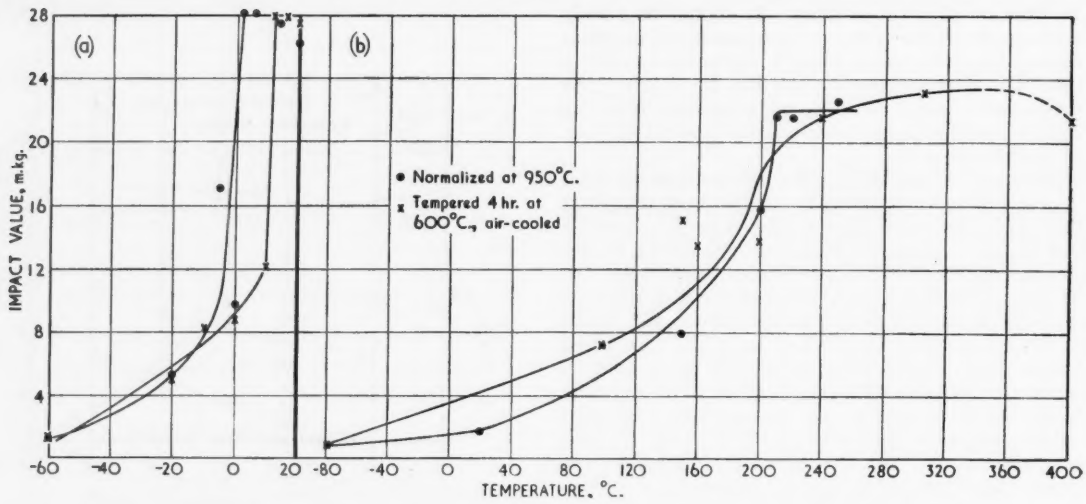

Fig. 8—Impact values at various temperatures of (*a*) 1% nickel alloy 79*AF*1 and (*b*) 3% nickel alloy 36*AF*2, normalized at 950° C. and tempered (after normalizing) 4 hr. at 600° C., air-cooled

factors governed the overall effect of tempering. The first factor, presumably associated with the traces of impurities present in the alloys, causes the transition temperature to be raised. The second factor, *viz.*, the relief of the lattice distortion inherent in the α_2 structure, causes the transition temperature to be lowered. The magnitude of the effect due to the first factor is considerable, the rise in transition temperature of the 2% nickel alloy being 70° C., and in this case it is considered that there was not a significant amount of α_2 structure. This effect might increase with nickel content, which would be consistent with the influence of nickel in accentuating the sensitivity of the alloys to traces of certain impurities that give rise to grain-boundary weakness. The magnitude of the effect due to the second factor would increase with the degree of distortion of the α_2 structure, *i.e.*, with nickel content. There was a reversal in the result of tempering of the 2–5% nickel alloys at the composition (about 3% of nickel) for which the two opposing factors had equal effects, and the result was that the transition temperature was lowered by increasing nickel content in the range 2–5% for alloys in the tempered condition.

The appearance of intergranular brittleness in the normalized material could be due to segregation of impurities at grain boundaries, a sensitivity to which is produced by the presence of nickel. Tempering at 600° C. might be expected to result in greater segregation of impurities at grain boundaries and greater weakening than would occur on normalizing at 950° C., which would account for the rise in transition temperature due to the first factor mentioned. It is of interest that although this effect could be detected in material that already had weak grain boundaries it was not pronounced enough to produce intergranular brittleness in the 1% nickel alloy, the grain boundaries of which remained stronger than the cleavage planes. It is presumably necessary to attain a threshold degree of segregation of harmful impurities at grain boundaries before fracture will occur there.

The marked tendency of the untempered α_2 structure in the iron–manganese and iron–nickel alloys to give poor notched-bar impact properties is noteworthy.

Effects of Small Amounts of Oxygen or Carbon in 1% Nickel Alloys

These 1% nickel alloys contained the stated impurities of oxygen or carbon and are useful in providing indications of the cause of the somewhat unexpected mechanical properties of the normal series of alloys, and of the effect of small amounts of certain elements. The oxygen-containing alloy was tested in the normalized condition only, the carbon-containing alloys in both the normalized and tempered conditions.

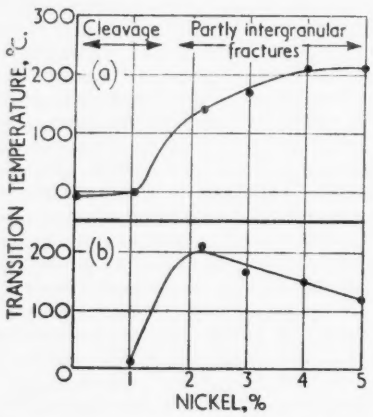

Fig. 9—Impact transition temperatures of iron–nickel alloys (*a*) normalized at 950° C., (*b*) tempered 4 hr. at 600° C., air-cooled

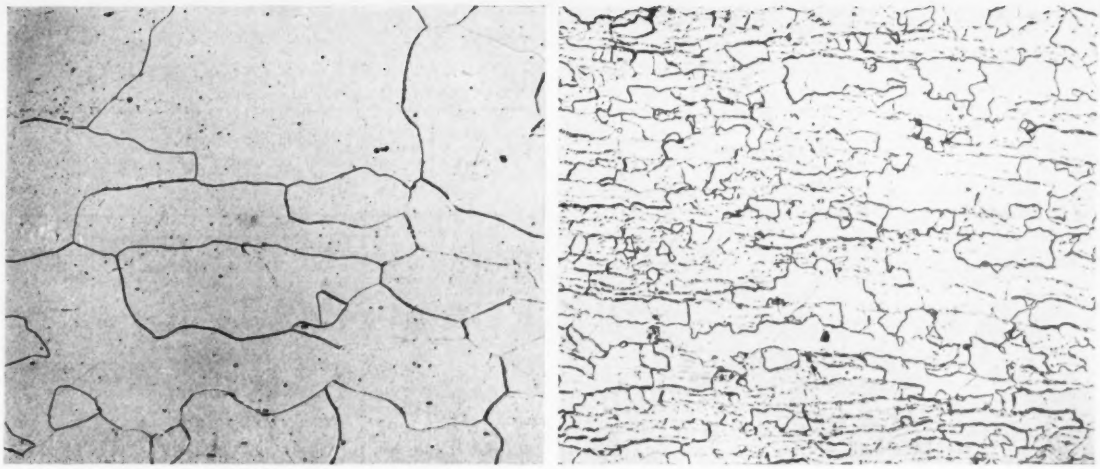

Fig. 10—Microstructure of normalized (*a*) 2% and (*b*) 4% nickel alloys × 150

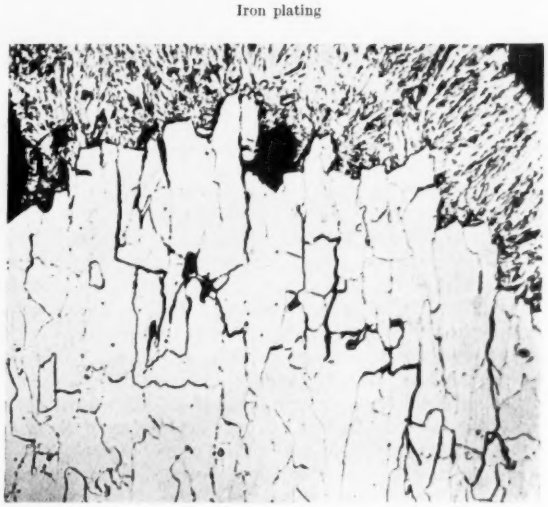

Fig. 11—Section through fracture of tensile test piece of 5% nickel alloy tested at −196° C., showing intergranular cracks and fracture × 250

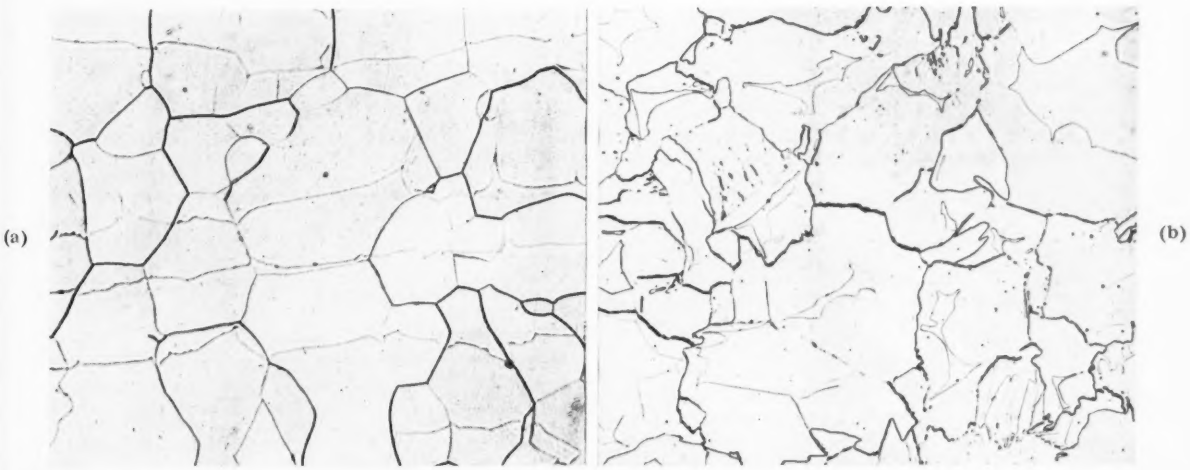

Fig. 12—Microstructure of 1% molybdenum alloy (*a*) normalized at 950° C.; (*b*) water-quenched from 950° C. × 150

Table IV

TENSILE PROPERTIES AT LOW TEMPERATURES OF IRON–1% NICKEL–OXYGEN OR –CARBON ALLOYS

Normalized at 950° C., Normalized and Tempered for 4 hr. at 600° C., and Air-Cooled

Mark	Alloy	Temp. of Test, °C.	Upper Yield Point, tons/sq. in.	Lower Yield Point, tons/sq. in.	0·2% Proof Stress, tons/sq. in.	0·5% Proof Stress, tons/sq. in.	U.T.S., tons/sq. in.	Elongation on $4\sqrt{\text{Area}}$, %	Reduction in Area, %	Fracture Stress, tons/sq. in.	Fracture
	Normalized at 950° C.										
79AF1	1·00% Ni	18	9·3	9·2	9·3	9·4	19·1	54	87	...	Ductile (cup and cone)
		− 73	15·4	13·6	14·8	14·1	22·8	65	86	...	" " "
		−196	...	...	39·0	40·2	46·4	9	9	51·0	Cleavage
34AF3	1·1% Ni + 0·01% O_2	21	9·2	9·2	9·2	9·2	17·7	48	85	...	Ductile (cup and cone)
		− 73	15·8	14·8	15·8	15·7	19·7	~20	11	23·7	Intergranular + cleavage
		−196	...	...	...	...	29·6	0	0	29·6	" "
73AF2	1·02% Ni + 0·005% C	20	...	...	10·5	11·5	19·8	55	88	...	Ductile (cup and cone)
		− 73	19·0	16·0	17·9	16·6	23·4	67	88	...	" " "
		−196	42·8	42·8	40·7	42·3	45·9	7	7	49·8	Cleavage
62AF1	0·97% Ni + 0·013% C	20	11·7	10·7	11·1	11·1	19·6	52	88	...	Ductile (cup and cone)
		− 73	15·5	14·0	15·1	14·6	24·0	66	88	...	" " "
		−196	...	...	39·7	40·7	45·7	7	7	48·8	Cleavage
	Normalized at 950° C., Tempered 4 hr. at 600° C., Air-Cooled										
79AF1	1·00% Ni	18	9·0	8·9	9·0	9·5	18·4	55	88	...	Ductile (cup and cone)
		− 73	15·7	13·5	14·3	13·5	22·5	65	87	...	" " "
		−196	...	...	36·5	37·4	41·2	4	3	42·5	Cleavage
73AF2	1·02% Ni + 0·005% C	19	12·7	11·0	10·9	11·0	19·4	55	90	...	Ductile (cup and cone)
		− 73	17·1	16·0	16·6	16·0	23·2	69	90	...	" " "
		−196	39·7	38·8	38·4	39·5	46·5	26	22	58·2	Cleavage, slight necking
62AF1	0·97% Ni + 0·013% C	20	10·7	10·2	10·3	10·3	19·0	58	89	...	Ductile (cup and cone)
		− 73	14·3	13·9	14·3	14·3	23·2	70	88	...	" " "
		−196	...	...	38·0	39·0	45·8	15	12	50·9	Cleavage

If the results of tensile tests (Table IV) on the 1% nickel, 0·01% oxygen alloy are compared with those of the high-purity 1% nickel alloy, it is seen that the presence of oxygen has little effect on the ductility at room temperature, but has caused a considerable reduction at − 73° C. The yield stress is little affected by the presence of the oxygen at room temperature or − 73° C., but the tensile strength at these temperatures is 2–3 tons/sq. in. lower. The effect of oxygen in weakening the material is demonstrated by the low fracture stress at − 73° and − 196° C., the test pieces at both temperatures failing in a brittle manner with predominantly intergranular fracture. The decrease in fracture stress with increase in temperature of test indicates that the strength at the boundaries decreases with rise in temperature at least over this range. The occurrence of brittle fracture at − 73° C. in this alloy shows that the tough-to-brittle transition in tension has been raised very much by an increase in oxygen content. This effect of oxygen is also observed in the notched-bar impact test, the transition temperature being exceptionally high at 290° C., and the brittle test pieces having mainly intergranular fractures.

The presence of 0·005% and 0·013% of carbon in a 1% nickel alloy in the normalized condition had little effect on the tensile properties (Table IV). The main effect of tempering at 600° C. was to cause a lowering of the transition temperature in tension, as evidenced by the enhanced ductility at − 196° C. The considerably higher fracture stresses at − 196° C. are a reflection of the improved ductility.

The transition temperatures in impact of these alloys in the normalized and the tempered conditions are given in Table V. Both alloys had lower transition temperatures than the high-purity nickel alloy in each condition. The transition temperatures of one of these alloys in the two conditions of heat-treatment are exceptionally low. This result is considered to be well outside errors arising from experimental variables and, although the effect is not understood, these results are included as further evidence of the sensitivity of iron–nickel alloys to small amounts of certain elements.

Table V

TRANSITION TEMPERATURE IN IMPACT OF 1% NICKEL, 1% NICKEL–OXYGEN, AND 1% NICKEL –CARBON ALLOYS

Mark	Composition	Transition Temperature, °C.	
		Normalized at 950° C.	Normalized, Tempered at 600° C. for 4 hr., Air-Cooled
79AF1	1% Ni	−3	+11
34AF3	1% Ni + 0·01% O_2	+290	...
73AF2	1% Ni + 0·005% C	−42	−52
62AF1	1% Ni + 0·013% C	−17	−10

Consideration of Results

A significant feature revealed by this investigation into the effect of nickel on high-purity iron has been the appearance of intergranular brittleness with a resultant deterioration in properties as the nickel content increases from 1% to 2%, as shown by a fall in fracture stress at − 196° C. and a rise in impact transition temperature. It is known that sulphur can cause brittleness in nickel and that the effect can be removed by the addition of such elements as magnesium. The sulphur contents of the iron–nickel alloys

Table VI

COMPARISON OF PROPERTIES OF 3% SILICON AND 3% NICKEL ALLOYS

Alloy	Impact Transition Temperature, ° C.	Yield Stress at 170° C., tons/sq. in.	Fracture Stress at − 196° C., tons/sq. in.
3% Si	164	19	24½
3% Ni	170	<12½	39

under discussion are in the range 0·004–0·0049%, and the possibility that this amount of sulphur is responsible in at least some degree for the grain-boundary weakness cannot be ignored. Alloys having higher sulphur contents of 0·01% with and without manganese present were made, but they were very brittle and cracked on rolling. No method is available for producing iron–nickel alloys with lower sulphur contents unless considerable effort is spent in desulphurizing the base iron. Previous experience has shown that weakness at the grain boundaries is likely to be associated with the presence of certain impurities, such as nitrogen, phosphorus, and oxygen. The nitrogen and phosphorus contents of the alloys were 0·001% or less, and it is considered hardly likely that such small amounts of these elements could account for the large effect observed. It has been established[2] that oxygen in amounts greater than about 0·003% in iron made in the 25-lb. induction furnace gives rise to grain-boundary weakness which becomes increasingly severe as the oxygen content is raised further, resulting in a raising of the transition temperature in impact and a progressive lowering of the fracture stress in tension at — 196° C. The oxygen contents of the 2–5% nickel alloys at 0·0014–0·0019% were below the level at which intergranular brittleness is first encountered in iron made in the same way. Nevertheless, whereas the 1% nickel alloy containing 0·0019% of oxygen was free from intergranular brittleness, a similar alloy with 0·01% of oxygen was considerably more brittle than the 2–5% nickel alloys. The grain boundaries of this alloy were in fact so weak that it broke with a brittle fracture and comparatively little deformation in tension, even at — 73° C.; its impact transition temperature was also exceptionally high. The brittleness in this alloy had about the same degree of severity as that produced by 0·05% of oxygen in iron free from nickel. The oxygen content necessary to produce comparable degrees of brittleness is reduced by about five times by 1% of nickel, and this, together with the appearance of intergranular brittleness in iron–nickel alloys at a lower oxygen content than in iron, suggests that nickel increases the effectiveness of oxygen in promoting grain-boundary weakness. If this is so, the somewhat sharp change in properties between 1% and 2% of nickel is consistent with the effect obtained in iron–oxygen alloys, where small increments in oxygen content above the value causing the first appearance of grain-boundary failure resulted in a more or less sharp fall in brittle-fracture stress and rise in impact transition temperature.

Whereas the transition temperatures in impact of the iron–nickel and iron–silicon alloys containing 2–5% of alloying element were in the same range, the iron–nickel alloys were ductile in tension down to at least — 73° C., but the iron–silicon alloys were not fully ductile below 200° C. For example, the relevant properties of the 3% silicon and 3% nickel alloys are compared in Table VI. Since the impact transition temperatures were approximately the same at 170° C. and the yield stress of the 3% silicon alloy was much higher than that of the 3% nickel alloy, the brittle-fracture stress at 170° C. of the 3% silicon alloy must have been much higher than that of the 3% nickel alloy. The reverse is true of the fracture stresses at — 196° C., however, so that as the temperature is raised to 170° C., either the brittle-fracture stress of the silicon alloy rises much more than that of the nickel alloy or the brittle-fracture stress of the nickel alloy is substantially decreased. Although some evidence has already been given to show that the brittle-fracture stress of the iron–silicon alloys is raised a little with increase in temperature, it appears probable that the brittle-fracture stress of the nickel

Table VII

CHEMICAL ANALYSES AND GRAIN SIZES OF IRON–CHROMIUM AND IRON–MOLYBDENUM ALLOYS

Element, %	38*AF*3 (1% Cr)	38*AF*2 (2% Cr)	38*AF*1 (3% Cr)	37*AF*3 (4% Cr)	37*AF*2 (5% Cr)	39*AF*1 (0·5% Mo)	40*AF*2 (1% Mo)	40*AF*1 (2% Mo)	39*AF*3 (3% Mo)	39*AF*2 (5% Mo)
Carbon	0·0028	0·0038	0·0046	0·0042	0·0030	0·0022	0·0024	0·0025	0·0017	0·0012
Silicon	0·003	0·003	0·004	0·003	0·014	0·003	0·003	0·002	0·003	0·002
Manganese	<0·005	n.d.	n.d.	n.d.	n.d.	<0·005	n.d.	n.d.	n.d.	n.d.
Sulphur	0·0048	0·0045	0·0048	0·0039	0·0042	0·0052	0·0048	n.d.	0·006	0·0051
Phosphorus	<0·001	n.d.	n.d.	n.d.	0·001	0·0013	n.d.	n.d.	n.d.	0·0012
Nickel	0·006	n.d.	n.d.	n.d.	0·005	0·006	n.d.	n.d.	n.d.	0·005
Chromium	1·20	2·32	3·37	4·12	5·58	0·001	n.d.	n.d.	n.d.	0·001
Copper	0·003	n.d.	n.d.	n.d.	0·003	0·005	n.d.	n.d.	n.d.	0·005
Aluminium	0·001	0·001	0·001	0·001	0·0015	0·006	0·003	0·005	0·006	0·005
Molybdenum	n.d.*	n.d.*	n.d.*	n.d.*	n.d.*	0·50	1·10	2·16	2·95	5·01
Oxygen	0·002	0·0024	0·0021	0·0032	0·0022	0·0016	0·0015	0·0008	0·00076	0·0012
Nitrogen	0·0008	0·0014	0·0012	0·0013	0·0016	0·0011	0·001	0·00094	0·0011	0·001
Hydrogen	← <0·000005 →									
A.S.T.M. grain size	2	2	4	4	5–6	4	2	4	2	2–3

n.d. = not determined
n.d.* = not determined but no molybdenum was detected spectrographically in the Swedish-iron base or the chromium from which these alloys were made

alloy falls considerably at temperatures above — 196° C., in view, for example, of the fact that such a decrease has been shown to occur in the 1% nickel, 0·01% oxygen alloy (*see* Table IV).

This argument assumes that the effect of the difference in rate of straining in the tensile and impact tests on the yield point is the same or not very different for both materials.

IRON–CHROMIUM AND IRON–MOLYBDENUM ALLOYS

The chemical analyses and grain sizes of alloys containing up to 5% of chromium and molybdenum are given in Table VII.

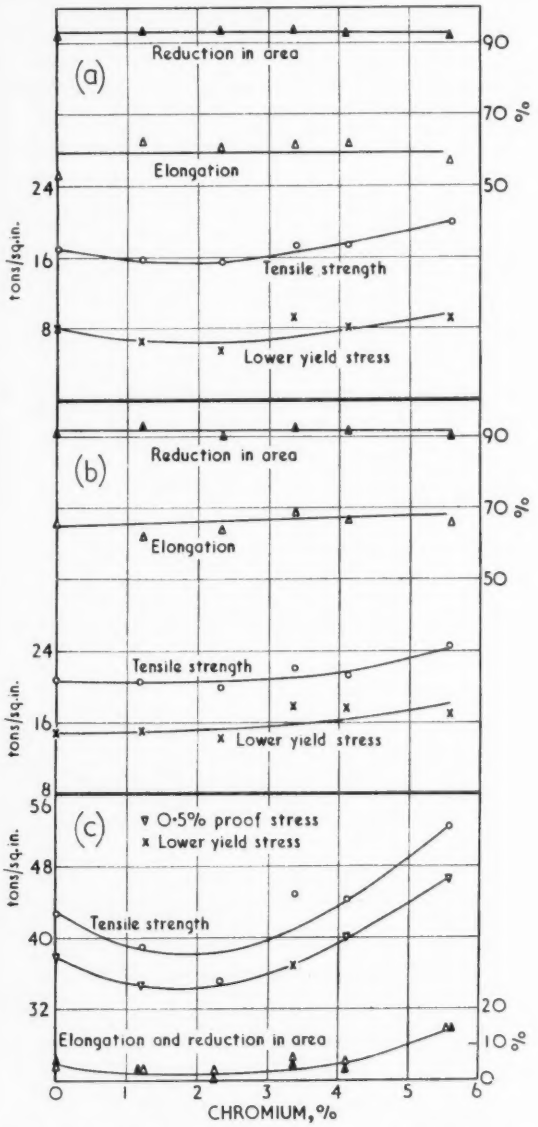

Fig. 13—Tensile properties at (*a*) room temperature, (*b*) — 73° C., and (*c*) — 196° C. of iron–chromium alloys normalized at 950° C.

Microstructures

The microstructure of the iron–chromium alloys consisted of polygonal grains, the size of which decreased somewhat as the chromium content increased. The grain size of all the molybdenum alloys was substantially constant and the microstructure of the 2–5% alloys was normal in consisting of polygonal grains. The 0·5% and 1% alloys had a microstructure similar to that shown in Fig. 12*a*, from which it will

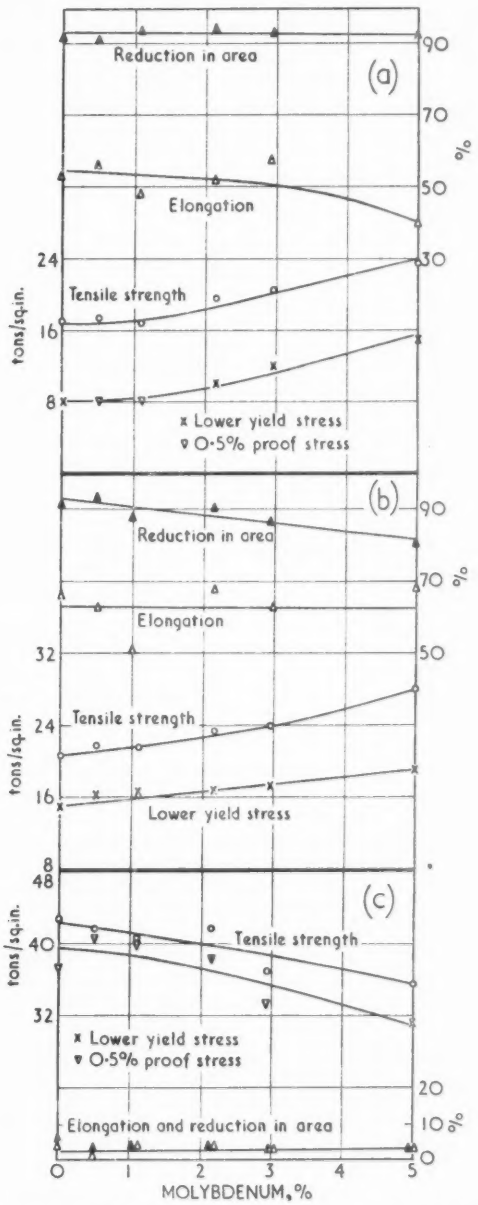

Fig. 14—Tensile properties at (*a*) room temperature, (*b*) — 73° C., and (*c*) — 196° C. of iron–molybdenum alloys normalized at 950° C.

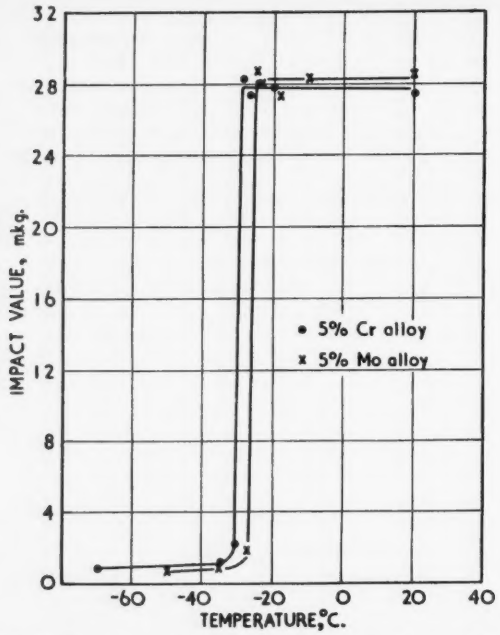

Fig. 15—Impact values at various temperatures of 5% chromium alloy 37*AF2* and 5% molybdenum alloy 39*AF2* normalized at 950° C.

be seen that in addition to the main grain boundaries there are fainter ones forming their own substructure. This type of microstructure is consistent with these two alloys having been in the duplex ($\alpha + \gamma$) region during heat-treatment at 950° C. followed by an intermediate rate of cooling such as air-cooling. By preserving the grain-boundary positions that existed at the elevated temperature and minimizing diffusion during cooling, water-quenching gave better defined microstructures (*see* Fig. 12*b*), which confirmed that these two alloys were in the ($\alpha + \gamma$) field at 950° C. There was no evidence in the microstructures of the normalized material that any of the 2–5% molybdenum alloys had been in the ($\alpha + \gamma$) field at 950° C.

The fact that the 0·5% and 1% molybdenum alloys had been heat-treated inside the ($\alpha + \gamma$) field, whereas the remaining molybdenum alloys had been heat-treated in the α field, does not seem to have had any effect on the properties.

Tensile Tests

The tensile properties at room temperature, — 73°, and — 196° C., are given in Figs. 13 and 14. The results for a high-purity iron are again included for the purpose of comparison. As seen from Fig. 13*a* and Fig. 14*a*, chromium had only a slight effect on the yield stress and tensile strength at room temperature, but molybdenum had a more pronounced effect in raising both these properties. The elongation and reduction in area were unaffected as the chromium content was raised, and the same is probably true of molybdenum, because although the elongation seemed to be lowered on increasing the molybdenum content from 3% to 5% no great weight is attached to this single result, as the same thing does not occur at — 73° C.

At — 73° C., the general effect of chromium and molybdenum on the tensile properties, shown in Figs. 13*b* and 14*b*, was similar to that at room temperature. There is a suggestion in Fig. 14*b* of a small decrease in reduction in area with increasing molybdenum content. A definite yield accompanied by a drop in load occurred in all the iron–chromium alloys at room temperature and — 73° C., but this occurred in only two molybdenum alloys at room temperature, although lowering the temperature of test to — 73° C. caused it to appear in all the alloys. The effect of decreasing the temperature to — 73° C. was to raise the yield stress, tensile strength, and elongation.

At — 196° C. (*see* Figs. 13*c* and 14*c*) all the chromium and molybdenum alloys failed in a brittle manner with mainly cleavage fracture. Traces of intergranular failure were seen on careful examination under a low-power binocular microscope with 1% and 4% chromium alloys and 0·5%, 2%, 3%, and 5% molybdenum alloys, and a slightly larger amount in the 2% chromium and 1% molybdenum alloys. The trace of intergranular failure in the chromium alloys had not generally affected the fracture stress, although the 2% alloy has a somewhat lower value than would be expected; in the molybdenum series the fracture stress did not appear to have been affected in the alloys with the larger amount of intergranular failure. It is considered that the traces of intergranular fracture in the chromium and molybdenum alloys can almost be neglected. The yield-stress and tensile-strength curves of the iron–chromium alloys at — 196° C. are of a similar shape to those at the higher temperatures of test, there being a minimum at about 2% of chromium. The elongation and reduction in area values also tended to rise for chromium contents above 2%. The ductility of the molybdenum alloys remained low at all molybdenum levels and there was a general decrease in yield stress and tensile strength with increasing molybdenum content.

Charpy Impact Tests

Impact values at various temperatures covering the tough-to-brittle transition were determined on each material and the impact-value/temperature curves for the 5% chromium and 5% molybdenum alloys are given in Fig. 15, these being typical of

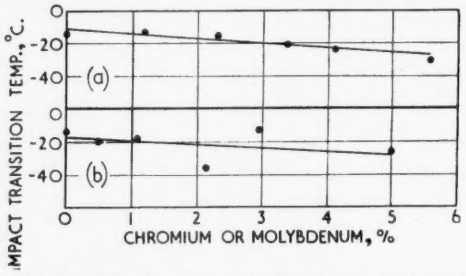

Fig. 16—Impact transition temperatures of (*a*) iron–chromium and (*b*) iron–molybdenum alloys normalized at 950° C.

curves obtained for each alloy. The transition from tough to brittle fracture was sharp in all the alloys. The transition temperatures are plotted in Fig. 16. The effect of chromium and molybdenum up to 5% was to cause a slight lowering of the impact transition temperature. The fractures of the impact test pieces that broke in a brittle manner were almost completely cleavage, but there was a trace of intergranular fracture in each case, as in the tensile specimens tested at — 196° C.

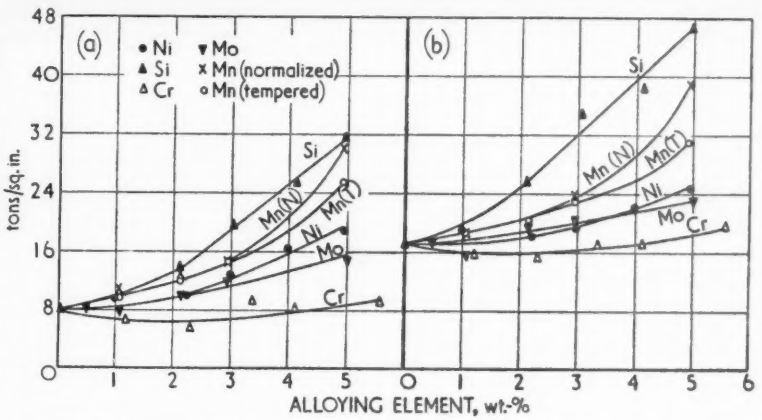

Fig. 17—Variation of (*a*) lower yield or 0·5% proof stress and (*b*) tensile strength with weight percentage of alloying elements in iron

Consideration of Results

Additions of up to 5% of chromium or molybdenum in iron exert a small strengthening effect without impairing the extremely high ductility at room temperature and — 73° C., which is in some contrast to the iron–manganese and iron–nickel alloys, in which a distinct decrease in ductility and a larger increase in yield stress and tensile strength are obtained in both the normalized and tempered conditions as the manganese or nickel content is raised. It is significant that chromium in amounts up to 2% has a distinct softening effect on iron at room temperature but is not so evident at — 73° C. This has also been shown to occur in the work of Edwards, Phillips, and Jones,[7] Lacy and Gensamer,[8] and Austin,[9] and no explanation can be given to account for this unusual effect. Lacy and Gensamer attribute this to the effect of chromium on small amounts of carbon, oxygen, or nitrogen as, for example, in its role as a deoxidizer. This is hardly likely to be the case, however, as they regard the treatment given to their irons in wet hydrogen as sufficient to remove carbon and nitrogen to such a low level that they would have no effect on the ageing characteristics of the iron. Furthermore, it has since been shown[2] that the tensile properties of iron at room temperature are not affected by oxygen in much larger proportions than was likely to be present in their materials. The explanation of Lacy and Gensamer for the unusual effect of up to about 2% of chromium on the tensile strength of iron is therefore considered to be unlikely.

It is of interest that the first additions of chromium caused a similar lowering of yield stress and tensile strength when the alloys were brittle, *i.e.*, at — 196° C., to that obtained at the higher temperatures of test when they were fully ductile.

The small lowering of transition temperature in impact caused by chromium could be accounted for either by a small progressive inferiority of yield stress or superiority of fracture stress at these temperatures. The small drop in impact transition temperature on adding molybdenum could be accounted for by a slightly larger increase in fracture stress over this temperature range than the increase in yield stress indicated by the tensile results at room temperature and — 73°C. Thus, the decrease in fracture stress at — 196° C. with increasing molybdenum content is not maintained at higher temperatures.

DISCUSSION

Some comment can be made on the factors affecting the properties in the ductile and brittle condition of the single-phase solid solutions dealt with in this paper, in order to emphasize the complexity of the effects obtained.

Dealing first with the alloys in the ductile condition, a comparison can be made of the respective strengthening effects of silicon, nickel, manganese, chromium, and molybdenum on high-purity iron using the present results and those obtained previously[1] for iron–manganese alloys in the normalized and tempered conditions. For this purpose, the proof stresses and tensile strengths at room temperature have been chosen for the nickel, manganese, chromium, and molybdenum alloys, and at 200° C. for silicon alloys, because this was the lowest temperature at which all the alloys were ductile. The proof stresses and tensile strengths of the tempered iron–nickel alloys have not been included in the comparison, as they were almost identical with those in the normalized condition.

Figures 17*a* and *b* show that the strengthening effect of these elements as given by the 0·5% proof stress and tensile strength increases in the order chromium, molybdenum, nickel, manganese, silicon. The rate of increase of strength rises with the weight percentage of alloying element, with the exception of chromium at the lower percentages. This is the same order as that given by Bain[10] and is similar to that given by Lacy and Gensamer[8] who, however, found that the strengthening effect of molybdenum was distinctly larger than that of nickel. In Fig. 18 the proof stress and tensile strength are plotted against the atomic percentage of each alloying element. The order of the elements giving increasing strengthening effect as judged by tensile strength, at least up to 3 at.-%, is chromium, nickel, silicon, manganese, and molybdenum, which is similar to that found by Lacy and Gensamer, except that silicon and molybdenum are interchanged. This difference may possibly be

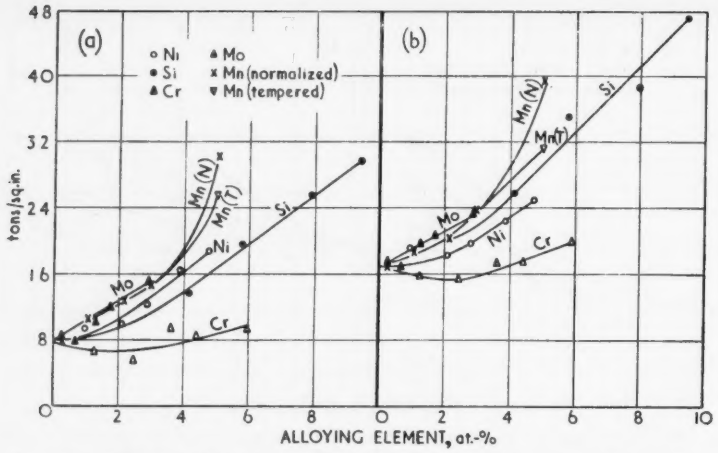

Fig. 18—Variation of (*a*) lower yield or 0·5% proof stress and (*b*) tensile strength with atomic percentage of alloying elements in iron

accounted for partly by the different grain sizes of the materials used in the two investigations, the grain sizes of the iron–silicon alloys used in the present work being considerably coarser than those of Lacy and Gensamer. It should be noted from Fig. 18 that the order of the elements giving increasing strengthening effect based on the proof-stress results is slightly different from that based on the tensile-strength results, the positions of nickel and silicon being reversed. No great weight is attached to this because in work on copper and silver alloys now proceeding at the National Physical Laboratory it is being found that proof stress is very susceptible to differences in previous history, whereas tensile strength for a given grain size is little affected, presumably because the factors governing the precise stage at which plastic deformation starts are no longer operative after considerable plastic work has occurred. Thus, tensile strength, although not a basic parameter, may well be more amenable to correlation with the factors determining the strengthening effect of an alloying element because of its insensitivity to certain details of history that have a large effect on the proof stress.

A theoretical treatment by Mott and Nabarro[11] concluded that the yield stress of a solid solution should be proportional to the square of the lattice distortion and to the concentration of solute. Frye and Hume-Rothery's work[12] on the strengthening of silver alloys by various alloying elements showed that the ultimate Meyer hardness was proportional to the square of the change in lattice parameter caused by given atomic percentages of alloying elements from the same period of the Periodic Table, but the slope of the straight line obtained was different for elements from different periods. Norbury[13] had earlier found that the Brinell hardness of a number of alloys of copper of given atomic concentration was approximately proportional to the difference in atomic volumes of the solvent and solute elements. That the hardening of binary alloys of ferrite is also linked up with the distortion of the lattice was suggested by Austin,[9] and that there is some such connection in the alloys dealt with here is shown in Fig. 19, where the change in lattice parameter of iron caused by various atomic percentages of chromium, molybdenum, nickel, manganese, and silicon is plotted against the tensile strength. It will be seen that a linear relation exists between these two quantities for alloys of silicon (which contracts the lattice), manganese in the tempered condition, and molybdenum (both of which expand the lattice), whereas for the iron–nickel alloys there is a change in slope above 2 at.-% and the iron–chromium alloys show a softening effect at the lower percentages as mentioned earlier. The change in the curve for the iron–nickel alloys above 2 at.-% is probably due to the presence of α_2, the distortion of which has not been relieved sufficiently by tempering at 600° C. In this connection it will be remembered that, from the change in transition temperature that occurred on tempering, the α_2 structure was thought not to be apparent at 1% or 2% of nickel, but present at 3% of nickel and above. The behaviour of the iron–nickel alloys is confirmed by the departure from linearity of the curve for the untempered iron–manganese alloys, which is almost certainly due to the presence of α_2, particularly in the 5% manganese alloy.

It could be argued from this that the type of distortion (probably local) that affects the properties is not necessarily given by the change in lattice parameter. This seems to be reasonable in view of Frye and Hume-Rothery's finding[12] on ternary alloys of silver with cadmium, indium, tin, and antimony that the hardness depends on the sum of the effects due to two additions separately rather than on the mean lattice distortion. The type of distortion obtained in α_2 structure, which gives rise to line broadening in the X-ray diffraction pattern, results in a much larger increase in strength than would be expected from change in lattice parameter only, and this indicates that a more precise definition of the term 'lattice distortion' may be necessary before the effects of alloying elements on the mechanical properties of a metal can be elucidated. It is also relevant to call attention to the very large effect on the properties of retaining in solution small amounts (up to 0·25 at.-%) of an interstitial element such as carbon,[3] where it has not yet proved possible to determine the precise type of distortion involved in the solid solution.

It can be argued that lattice distortion is not the main factor governing the strengthening effect observed when it is considered that the increase in strength of the iron–silicon alloys is large for only a small difference in lattice parameter, whereas with molybdenum as the solute the lattice distortion is considerably larger for the corresponding atomic

percentage but the strengthening effect is much smaller. This is in spite of the fact that the iron–silicon alloys were coarser-grained than the iron–molybdenum alloys, and the difference would be expected to be larger for similar grain sizes. Furthermore, considerations of lattice distortion appear to throw no light on the softening obtained with the first 2% of chromium. Other factors that have been suggested as being involved are difference in valency between solute and solvent,[14] and electron/atom ratio[15]; in the present work, however, the number of valency electrons in any particular case is not known.

Considering now the factors affecting the properties in the brittle condition, the brittle-fracture stress of iron–silicon alloys and the effect this has on the transition from tough to brittle fracture in tension and notched-bar impact is dominated by the grain-boundary weakness that appears in the coarse-grained or normalized condition; the brittle-fracture stress reflects the grain-boundary strength rather than the cleavage strength. The reduction of the brittle-fracture stress of iron occurs by the addition of the first 1% of silicon, and there is little further effect when the silicon content is increased to 5%. The increase in transition temperature in tension and impact on increasing the silicon content from 1% to 5% can be explained by the considerable increase in proof stress, whilst the brittle strength at — 196° C. does not change appreciably. The fact that the intergranular brittleness could be removed in the 1% silicon alloy by making the material fine-grained by a cold-working and annealing treatment suggests that the tendency of the normalized material to break along grain boundaries could be due to a concentration of one or more impurities in these regions.

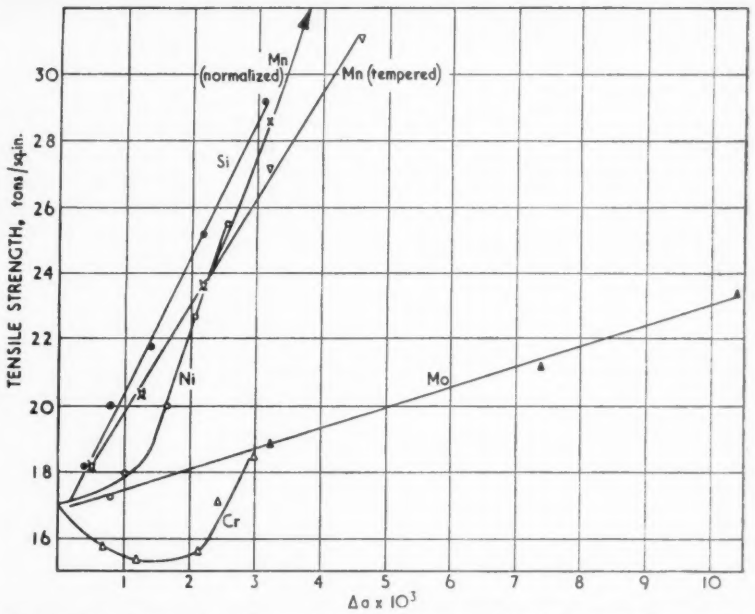

Fig. 19—Variation of tensile strength with change in lattice parameter caused by up to 5 at.-% of alloying elements in iron. The points on the curves represent at.-% up to 5

The effect of nickel on the properties of iron in the brittle range is complicated. Two factors appeared to influence its behaviour: the occurrence of intergranular weakness in those alloys containing more than 1% of nickel and the formation in some degree of α_2 structure. Alloys containing more than 1% of nickel had low fracture stresses in tension at — 196° C. and high transition temperatures in impact, whereas the 1% alloy which broke entirely by cleavage in the brittle condition had properties very similar to those of high-purity iron.

The appearance of intergranular weakness with consequent deterioration in properties of the 2–5% nickel alloys is likely to be an effect of segregated impurities which is accentuated by the presence of nickel. Sulphur, which was present to the extent of 0·004–0·005%, cannot be ruled out as a partial cause of the intergranular weakness, but direct evidence was obtained that when oxygen was added its effect in this direction was about five times as great as in nickel-free iron. It is feasible, therefore, that the deleterious effect of oxygen is more easily made evident in the presence of nickel than in the absence of nickel, *i.e.*, that oxygen in iron–nickel alloys could give rise to intergranular brittleness even when present to such a small extent as in the alloys examined, *viz.*, about 0·0015%.

The effect of tempering the iron–nickel alloys in modifying the trend of the impact transition temperature with increasing nickel content (*see* Fig. 9) could be explained on the basis of the relative effects produced by this treatment on the tendency to break along grain boundaries and the relief of the distortion due to α_2 structure. The result of this was that the transition temperatures in both tension and impact were lowered by increasing nickel content from 2% to 5% in the tempered condition, whereas in the normalized condition the transition temperature in impact was raised but that in tension was lowered.

Evidence is presented to show that a change in temperature can have a substantial effect on the brittle strength. In the 1% nickel, 0·01% oxygen alloy, the brittle-fracture stress decreased with rise in temperature, and it is likely that the same thing happens in plain iron–nickel alloys; on the other hand, there seems to be a slight rise in brittle-fracture stress in the iron–silicon alloys with rise in temperature. There is also a complicated effect of temperature in the iron–molybdenum alloys, the impact test results indicating that, although increasing the molybdenum

content caused a reduction in fracture stress at — 196° C., the reverse must be true at about — 20° C., assuming that the effect of strain rate on the yield stress is similar at all molybdenum levels.

Whatever the reason for the softening effect of the first additions of chromium when the alloys are tested in tension in the ductile range, it would appear that the same explanation might be applied to the similar decrease in yield stress and tensile strength (or fracture stress) of the alloys containing up to 2% of chromium when tested in the almost completely brittle condition at — 196° C.

Acknowledgments

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Effect of Heat-Treatment on the Brittleness of High-Purity Iron–Nitrogen Alloys

By B. E. Hopkins, M.Sc., and H. R. Tipler, B.Met.

SYNOPSIS

The results of tensile and notch-impact tests on two high-purity iron–0·01% nitrogen alloys at temperatures spanning the tough-to-brittle transition are discussed. The intergranular weakness encountered on adding nitrogen to iron may be almost entirely removed by furnace-cooling from the austenitic region, but its severity is enhanced by increasing the rate of cooling. Maximum embrittlement of the grain boundaries is developed well inside the α solid-solution field at 600–700° C., water-quenching from these temperatures resulting in remarkably low brittle-fracture stresses. This extreme brittleness could be caused by grain-boundary segregation of nitrogen, which tends to be dispersed at higher and lower temperatures. There is a linear rise with temperature in the brittle-fracture stress of the material in the most brittle condition over the wide range of —196° C. to room temperature. The microstructures of the alloys after various heat-treatments are discussed in the light of recently published work. 911

Investigations of the properties of high-purity iron and its alloys from the point of view of the transition from a tough to a brittle fracture are complicated by the occurrence of two types of failure in the brittle condition. In some iron alloys brittle fracture occurs solely by cleavage along simple crystallographic planes, whereas in other alloys or conditions of heat-treatment it occurs along grain boundaries, although failure is then usually accompanied by varying amounts of cleavage. Grain-boundary failure occurs when the grain-boundary strength is less than the cleavage strength and, even though the brittle fracture is only partly along the grain boundaries, grain-boundary strength rather than cleavage strength governs the properties.

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In work carried out at the National Physical Laboratory in recent years, intergranular brittleness has been encountered in a number of irons and iron alloys. Grain-boundary weakness had been found in iron on a number of previous occasions but it had never been investigated. It has now been established that oxygen is one cause of intergranular brittleness in iron, and the results of a detailed examination of this have been published.[1] However, other factors besides oxygen content may be responsible for intergranular brittleness. For example, low brittle-fracture stresses due to intergranular weakness can be produced in iron containing less than about 0·006% of carbon by water-quenching from either above or below the A_3 point, whereas in other conditions of heat-treatment brittle fractures entirely of the cleavage type are obtained.[2] In these materials grain-boundary weakness can be removed easily by heat-treatment. What appeared to be a similar type of intergranular brittleness has been encountered in a more severe form in iron–nitrogen and iron–phosphorus alloys. The present paper deals with a more detailed examination of the phenomenon in two high-purity iron–nitrogen alloys of about the same composition.

PREPARATION OF THE ALLOYS

Two alloys, 44*AF*1 and 44*AF*2, were made on a 25-lb. scale by a process described elsewhere.[3] Specially selected Swedish iron which had previously been oxidized by melting in air was remelted *in vacuo* and deoxidized with hydrogen dried by circulation through silica gel. Nitrogen was introduced into the iron by having a partial pressure of the gas above the melt during the deoxidation period. When deoxidation was complete, the hydrogen + nitrogen pressure was reduced to a few centimetres before pouring into a mild-steel mould.

The analyses of the alloys are given in Table I. Both alloys contained about 0·01% of nitrogen, and impurities were uniform and low in amount.

The ingots, which were 10½ in. long without the feeder head and 2¾ in. across the flats of the octagonal section, were hot-rolled to ⅝-in. dia. bars with intermediate reheating.

TESTING AND HEAT-TREATMENT

Tensile tests were carried out at room temperature, — 73°, and — 196° C. in a hydraulic testing machine

Table I

CHEMICAL ANALYSES OF TWO IRON–NITROGEN ALLOYS

Element, %	Alloy 44*AF*1	Alloy 44*AF*2
Carbon	0·003	$0·004_3$
Silicon	0·003	0·003
Manganese	<0·005	...
Sulphur	$0·005_8$	$0·003_6$
Phosphorus	0·001	...
Nickel	0·006	...
Chromium	0·001	...
Copper	0·005	...
Aluminium	0·001	0·004
Oxygen	$0·001_5$	$0·001_3$
Nitrogen	$0·009_4$	0·010
Hydrogen	<0·000005	

using 0·282-in. dia. specimens with a 1-in. gauge length and ½-in. B.S.W. threads; 0·226-in. dia. specimens were used at low temperatures when the material was in its most brittle condition. Proof stresses were measured using an extensometer[2] that is not damaged if a test piece breaks in a brittle manner before the proof stresses are reached.

Impact tests were carried out over a temperature range spanning the tough-to-brittle transition using 10-mm. square × 55 mm. long Charpy test pieces with an Izod **V** notch (2 mm. deep × 0·25 mm. root rad.). The tough-to-brittle transition temperature was taken as the temperature at which the impact value was half that in the fully tough condition.

The first series of experiments concerned the effect of rate of cooling (furnace-cooling, air-cooling, and water-quenching) from 950° C. on the tensile and impact properties of alloy 44*AF*1. Further tests were carried out on specimens of this alloy after furnace-cooling from 950° to 300° C. and then water-quenching to retain the nitrogen in solution, according to the solubility data of Dijkstra[4] and Borelius, Berglund, and Avsan.[5] Tests were also done on specimens that had been air-cooled or water-quenched from 950° C. and aged at room temperature for about 8 weeks. These heat-treatments were carried out on specimens sealed in evacuated silica tubes. The impact test pieces were notched immediately after heat-treatment, and the tests were completed the same day where it was desired to avoid ageing effects.

As a result of this work, further heat-treatments consisting of furnace-cooling from 1150° C., followed by various isothermal treatments, were done on tensile test pieces of alloy 44*AF*2. These treatments are dealt with in detail when describing the results.

The grain sizes of the alloys after the different heat-treatments are given in Table II.

Table II

GRAIN SIZES OF THE ALLOYS AFTER VARIOUS HEAT-TREATMENTS

Treatment	Grain Size A.S.T.M. Scale	Grains/mm.
F.C. from 950° C.	3	8
A.C. from 950° C.	4	12
W.Q. from 950° C.	5–6	19
F.C. from 950° to 300° C. and W.Q.	3	8
F.C. from 1150° C.	0	3

PROPERTIES AFTER COOLING FROM THE AUSTENITIC REGION

The effect of cooling rate from 950° C. on the tensile properties of alloy 44*AF*1 at room temperature, — 73°, and — 196° C. is apparent from the results given in Table III. In the ductile condition (*i.e.*, at room temperature and — 73° C.) sharp yield points with a drop in load were given by the specimens furnace-cooled or air-cooled, but water-quenching suppressed this yield behaviour. The effect of increasing the rate of cooling from 950° C. was to raise the yield (or proof) stress and tensile strength, the effect on the yield stress being more marked than that on the tensile strength. This effect of cooling rate is probably caused almost entirely by the decrease in grain size, and only to a very small extent by the

Table III

TENSILE PROPERTIES OF ALLOY 44*AF*1

Tensile Properties	Room Temperature					−73° C.					−196° C.				
	F.C. 950° to 300° C., W.Q.	F.C. 950° C.	A.C. 950° C.	W.Q. 950° C.	A.C. 950° C., Aged at Room Temp.	F.C. 950° to 300° C., W.Q.	F.C. 950° C.	A.C. 950° C.	W.Q. 950° C.	A.C. 950° C., Aged at Room Temp.	F.C. 950° to 300° C., W.Q.	F.C. 950° C.	A.C. 950° C.	W.Q. 950° C.	A.C. 950° C., Aged at Room Temp.
0·2% proof stress, tons/sq. in.	8·4	6·3	8·6	10·7	10	12·7	...	15·9	16·2	18·7	...	...	...	...	...
0·5% proof stress, tons/sq. in.	8·9	6·7	8·6	11·4	10	12·4	...	14·6	16·6	18·7	...	...	...	...	...
Upper yield stress, tons/sq. in.	...	6·7	8·6	...	10	12·9	22·7	16·8	...	18·7	...	...	...	...	...
Lower yield stress, tons/sq. in.	...	6·3	8·5	...		12·3	17·1	14·1	...		...	...	...	...	...
U.T.S., tons/sq. in.	19·5	16·0	19·5	19·9	18·3	22·7	22·8	22·6	24·2	19·9	35·6	36·2	28·1	18·4	33·6
Fracture stress, tons/sq. in.	...	...	...	...	...	...	...	...	28·1	23·1	35·6	36·2	28·1	18·4	33·6
Elongation, %	52	64	53	55	60	60	59	60	19	14	0	0	0	0	0
Reduction in area, %	92	94	86	90	93	71	92	77	14	13	0	0	0	0	0

retention of the 0·01% of nitrogen in solution by the faster rates of cooling. The yield stress and tensile strength at — 73° C. for the furnace-cooled condition were somewhat higher than expected, the yield stress being actually higher than for the air-cooled condition. This result has been confirmed beyond doubt, but the somewhat more rapid rise of yield stress and tensile strength with decrease in testing temperature for the furnace-cooled condition has not been investigated further. It may be significant that this is the only condition in which nitride needles were present. In the water-quenched condition the alloy gave a brittle fracture at — 73° C. although considerable elongation and reduction in area had taken place in the test piece; the material in this condition at — 73° C. was thus within the range of transition from tough to brittle fracture in tension, whereas in the air-cooled and furnace-cooled conditions the transition temperature range was below — 73° C. This is consistent with the behaviour of the material in tension at — 196° C. and in the impact tests.

At — 196° C. the alloy was completely brittle for all conditions of heat-treatment, the fracture stress showing a large decrease with increase in the cooling rate from the austenite region. The fractures were mixed cleavage and intergranular, and the proportion of a fracture that occurred along grain boundaries became larger as the fracture stress decreased. There is little doubt that the fracture stress was governed by the grain-boundary strength, *i.e.*, that brittle fracture started along the grain boundaries.

Furnace-cooling from 950° to 300° C. followed by water-quenching suppressed the normal yield point at room temperature, but a slight yield was obtained at — 73° C. The proof stresses at room temperature were higher after this treatment than after straight furnace-cooling, but the rate of rise of the proof stress with decrease in testing temperature was less than for any other condition tested, so that at — 73° C. the proof stresses were well below those for the plain furnace-cooled condition. At — 196° C. the fracture was completely brittle and consisted of mixed cleavage and intergranular types; the fracture stress was about the same as for the furnace-cooling treatment.

The main effects of ageing the air-cooled material at room temperature for 8 weeks were to raise the yield stress at room temperature and — 73° C. and to raise the fracture stress at — 196° C. It is also curious that although in the freshly air-cooled condition upper and lower yield points were obtained, after ageing there was no drop in load but only an arrest at the yield at both room temperature and — 73° C. The specimen tested at — 73° C. was brittle with no necking, so that the tensile strength was lower than if the specimen had necked. This single result indicates that the transition temperature range in tension had been raised by ageing, whereas the transition temperature in impact was lowered.

The effect of the heat-treatments at 950° C. on the behaviour of alloy 44*AF*1 in the Charpy impact test is shown by the impact-value/temperature curves given in Figs. 1*a* and *b* and the impact transition temperatures plotted in Fig. 2. The highest transition temperature was obtained by water-quenching; the effect of decreasing the rate of cooling from 950° C. was to lower the transition temperature progressively. Furnace-cooling from 950° to 300° C. followed by water-quenching to retain the nitrogen in solution gave a lower transition temperature than the simple treatment of furnace-cooling to room temperature. Ageing at room temperature for 8 weeks after air-cooling or water-quenching from 950° C. caused a lowering of the transition temperature, and this effect was larger for the initially water-quenched specimens.

The effects of increasing cooling rate from the austenitic condition in raising the impact transition temperature, and of ageing in lowering the transition temperature, are both the reverse of what happens in iron–carbon and iron–carbon–manganese alloys.[2] As might be expected from the high transition tem-

peratures compared with that of high-purity iron, the brittle fractures of the Charpy test pieces were all partly along the grain boundaries, the proportion of intergranular failure becoming larger as the transition temperature increased.

When interpreting differences in transition temperature in impact in terms of differences in yield stress and brittle-fracture stress, the two tensile parameters should ideally refer to the temperature range over which the mode of failure in the impact test changes from tough to brittle. A method of determining the brittle-fracture stress at such temperatures has not yet been developed, so that the fracture stresses obtained at the much lower temperature of — 196° C. must be relied on. Furthermore, the highest temperature adopted for tensile tests in this work was room temperature, which is substantially below the impact transition temperatures. Nevertheless, a knowledge of the trends of the yield stress at room temperature with variation in heat-treatment procedure is probably adequate for explaining the gross changes in impact transition temperature.

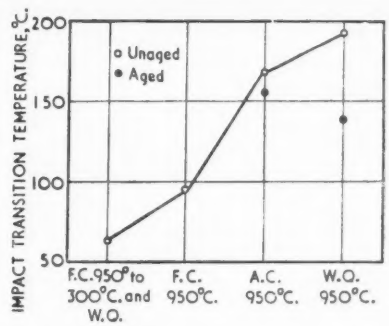

Fig. 2—Effect of heat-treatment on the Charpy impact transition temperature of alloy 44*AF*1

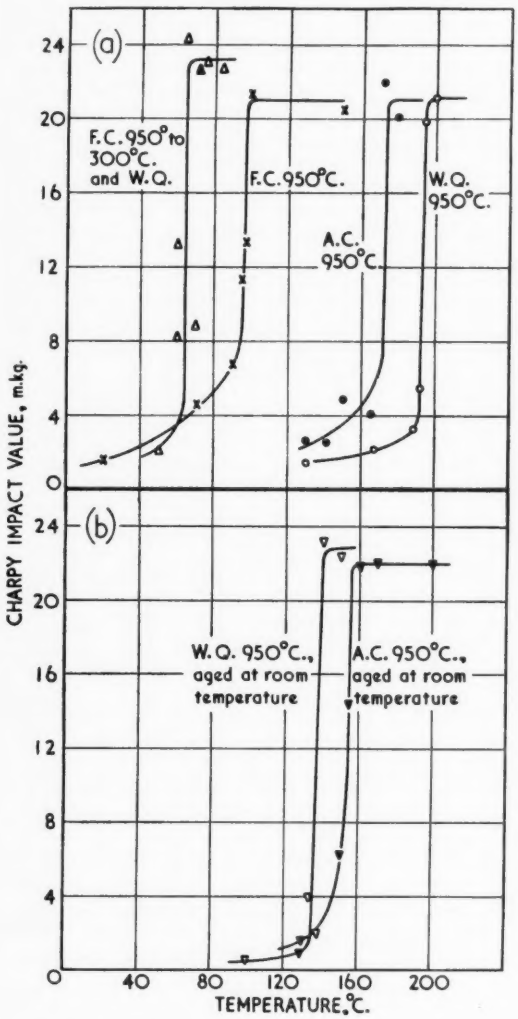

Fig. 1—Charpy-impact-value/temperature curves for alloy 44*AF*1 in various conditions of heat-treatment

The raising of the impact transition temperature by increasing the cooling rate from 950° C. must be due in part to the rise in the yield stress, but it is probably due mostly to the decrease in brittle-fracture stress. In fact, the magnitude of the decrease in brittle-fracture stress with increase in the cooling rate from the austenitic region is such that a larger effect on the transition temperature in impact might well have been expected. Furnace-cooling to 300° C. followed by water-quenching gave a higher proof stress than furnace-cooling to room temperature, so that the lower impact transition temperature after the former treatment was probably the result of a higher brittle-fracture stress in the temperature range covered by the impact tests, even though the brittle-fracture stresses at — 196° C. were about the same for the two treatments.

The drop in impact transition temperature of the air-cooled material as a result of ageing is attributed to the rise in brittle-fracture stress because the yield stress was also raised. Similar remarks probably apply to the effect of ageing on the water-quenched material, but tensile tests were not carried out in this case in the aged condition. This rise in brittle-fracture stress, *i.e.*, strengthening of the grain boundaries, might be ascribed to diffusion of nitrogen away from the grain boundaries at room temperature, as is later indicated to occur at, say, 300° C. The ageing of iron–nitrogen alloys is likely to be a complicated phenomenon because of the combined effect on the properties of precipitation of nitrides, removal of nitrogen from solution, and diffusion of nitrogen away from grain boundaries.

ISOTHERMAL EMBRITTLEMENT EXPERIMENTS

It is interesting that the lowering of brittle-fracture stress and raising of impact transition temperature with increasing cooling rate from the austenitic condition are the reverse of the effects obtained with iron–carbon and iron–carbon–manganese alloys. In the iron–nitrogen alloys the effect

Table IV

TIMES OF ISOTHERMAL TREATMENTS TO GIVE EQUAL DIFFUSION OF NITROGEN

Temperature, °C.	Time
1000	1 hr. 45 min.
900	20 min.
800	40 min.
700	1 hr. 36 min.
600	4 hr. 36 min.
500	16 hr. 42 min.
450	38 hr.
400	3 days 22 hr.
300	39 days 4 hr.

was accompanied by an easily recognizable increase in the proportion of the fracture occurring along grain boundaries and was undoubtedly a reflection of a decrease in the grain-boundary strength; in the iron–carbon and iron–carbon–manganese alloys the fractures were entirely of the cleavage type, and so it was the cleavage strength that was raised by increasing the cooling rate. The effect of cooling rate in the iron–nitrogen alloys was investigated further on the supposition that increasing it favours the retention of a condition existing at an elevated temperature. It was thus of interest to know whether there is a temperature range in which weakening of the grain boundaries takes place. This was investigated by determining the fracture stress at — 196° C. after various isothermal treatments.

Specimens of alloy 44*AF*2 were first heat-treated to obtain something near the highest fracture stress attainable by furnace-cooling from the austenitic region. So that no grain coarsening should occur during the subsequent isothermal treatments, a fairly high soaking temperature (1150° C.) was chosen. The specimens were then heated at temperatures from 300° to 1000° C., mainly at 100° intervals, for times that would give equal opportunity for diffusion of nitrogen, and then water-quenched. The ratios of the times of heating at different temperatures were calculated from the diffusion data given in the literature.* The actual times of heating, shown in Table IV, were chosen such that:

(i) At the highest temperatures they were not so short as to be unreliable because of any error in the estimate of the time taken by the specimens sealed in evacuated silica tubes to reach the furnace temperature.

(ii) They were many times larger than the theoretical estimate for diffusion of nitrogen from the middle of a grain to the boundary, knowing the grain size.

In view of the second consideration, the results represent equilibrium values.

The results of the tensile tests at — 196° C. on specimens treated in this way are shown in Fig. 3. If weakening of the grain boundaries is due to diffusion of nitrogen to these regions, and if the driving force for diffusion were unaffected by temperature, the fracture stress in these experiments would have been constant, giving a vertical straight line in Fig. 3. If the driving force for diffusion of nitrogen to the boundaries were affected by temperature in a uniform manner (*e.g.*, the lower the temperature the greater the tendency for diffusion to the boundaries) the results in these experiments would have fallen on a sloping line, the fracture stress being less at lower temperatures of diffusion. The results indicate that neither of these situations is true. The fracture stress of the material furnace-cooled from 1150° C. was 29·6 tons/sq. in., whereas the subsequent isothermal treatments gave a minimum value of about 5 tons/sq. in. for temperatures of 600–700° C. The strength of the grain boundaries after heating in this temperature range was thus remarkably low. The appearance of the fractures was consistent with the fracture stresses, the specimens with the lowest fracture stresses having fractures that were almost entirely intergranular with increasing proportions of cleavage as the fracture stress became higher. That the effect was not a permanent feature of the alloy but was readily modified by changing the heat-treatment is shown by the fact that specimens furnace-cooled from 1150° to 625° C. and water-quenched gave a fracture stress at — 196° C. of 6·7 tons/sq. in., which is very similar to the result obtained by heating at 600° C., whereas furnace-cooling to room temperature gave 29·6 tons/sq. in. This shows that the fracture stress of the alloy during furnace-cooling goes through the cycle indicated in Fig. 3.

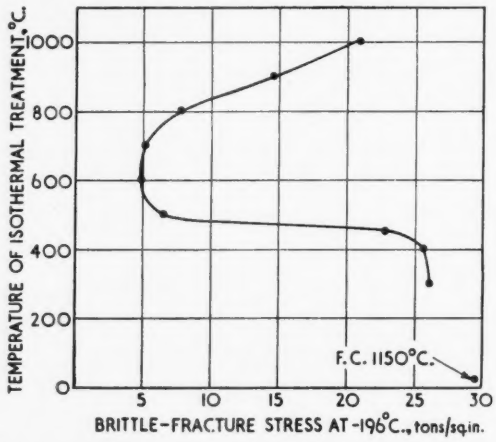

Fig. 3—Brittle-fracture stresses at — 196° C. of alloy 44*AF*2 F.C. from 1150° C. and subsequently heated at the temperature shown for times adjusted to give equal opportunity for diffusion of nitrogen, and then W.Q.

* Up to 900° C. the values given by Wert and Zener[6] ($D_0 = 1{\cdot}4 \times 10^{-3}$ sq. cm./sec. and $Q = 17{,}700$ cal./mole in the diffusion equation $D = D_0 e^{-Q/RT}$) have been used. Wert's values[7] are slightly different: $D_0 = 3 \times 10^{-3}$, $Q = 18{,}200$. Since this work was done, Fast and Verrijp[8] have published the values $D_0 = 6{\cdot}6 \times 10^{3}$, $Q = 18{,}600$. Their values are probably to be preferred because they covered the widest temperature range: 20–900° C. for α-iron. However, the ratios of the times at various temperatures to give equal diffusion are not very different whichever of these values are used. Bramley's value[9] of $D = 13{\cdot}5 \times 10^{-8}$ has been used at 1000° C. His value of D at 950° C. ($10{\cdot}8 \times 10^{-8}$) is similar to that of Fast and Verrijp ($6{\cdot}5 \times 10^{-8}$), so that there is some confidence in using Bramley's value at 1000° C., even though his data at lower temperatures (*e.g.*, 800° C.) are substantially different from those of more recent workers.

To determine the time required for the development of the extreme embrittlement encountered at about 600–700° C., a number of tensile specimens that had been furnace-cooled as before from 1150° C. were heated at 600° C. for progressively shorter times, and were then water-quenched and their brittle-fracture stresses at — 196° C. determined. The results are given in Table V. For the two shortest times the heating was done in a lead bath, and a furnace was used for the longer times. The shortest time (1 min.) is somewhat indefinite because of uncertainty in the time required for the specimens to reach the lead-bath temperature. The results show that a constant low fracture stress was obtained for all the times chosen, so that the embrittlement was substantially complete in a very short time (about 1 min.) at 600° C.

From the equation[10]

$$\bar{s}^2 = 2Dt$$

where $\bar{s}^2$ is the mean square of the component of the displacement of the atoms in a given time t and D is the diffusion coefficient, an estimate can be obtained of the time required for nitrogen atoms to diffuse from the middle of a grain to the boundary. The grain size of alloy 44*AF*2 after furnace-cooling from 1150° C. was 3 grains/mm., so that t is about 45 min. If it were necessary for most of the nitrogen to diffuse to the grain boundary before the lowest fracture stress of about 5 tons/sq. in. is reached, the calculated time is too long. It was noticed, however, that sub-grains were visible in the structure and, using an average figure of 10 sub-grains/grain on a section, the time for diffusion to the sub-grain boundaries is about 27 sec. This is consistent with the experimental finding that embrittlement occurs in 1 min. at 600° C., but in this case fracture might be expected to occur largely along the sub-grain boundaries. A considerable number of sections of fractures have been examined carefully under the microscope, particular attention being paid to cracks away from the fracture, and in no case could it be shown definitely that cracks were occurring along sub-grain boundaries, whereas in most cases there was little doubt that the main grain boundaries were involved.

A more likely explanation is that it is not necessary for the majority of the nitrogen atoms to reach grain boundaries before a minimum fracture stress is obtained. If only a fraction of the nitrogen atoms are required for this extreme embrittlement of the boundaries, from volume considerations these could be supplied from near the grain boundary, making the distance of diffusion involved much smaller. Since it is feasible that quite thin layers of nitrogen-rich material are sufficient to make the grain boundaries brittle, the time required for this segregation could be very small if the driving force for this directed diffusion were strong enough compared with the homogenizing effect of thermal vibrations.

This discussion is based on the supposition that the embrittlement is caused by segregation of nitrogen at the grain boundaries, the most straightforward assumption. However, the evidence for such segregation is not direct. A similar cooling-rate effect on the grain-boundary strength has since been found in iron–phosphorus alloys. These alloys are being investigated further and, because the little information available about the rate of diffusion of phosphorus in iron indicates that it is less than that of nitrogen, it may be easier to determine whether the embrittlement is consistent with diffusion of phosphorus atoms to the grain boundaries.

The brittle-fracture stress at — 196° C. of alloy 44*AF*2 after furnace-cooling from 950° C. was similar to that of alloy 44*AF*1 at about 39 tons/sq. in., with a grain size of about 8 grains/mm. This is higher than the value of 29·6 tons/sq. in. after furnace-cooling from 1150° C. which gave a grain size of about 3 grains/mm. This influence of grain size is a feature of intergranular brittleness, the amount of material available for segregation at the boundaries being larger per unit grain surface area with larger grain sizes. After furnace-cooling from 950° C. there was only a trace of grain-boundary failure, whereas it was distinctly more pronounced after furnace-cooling from 1150° C.

To determine the temperature-dependence of the fracture stress of alloy 44*AF*2 in the most brittle condition, tensile tests were carried out at temperatures up to 100° C. on the material after furnace-cooling from 1150° C. and water-quenching from 600° C. The results are shown in Fig. 4. The fracture stress increased linearly as the testing temperature was raised from — 196° C. to room temperature, but at 100° C: it was somewhat above the value that would be expected from the trend at lower temperatures. At 100° C., however, the results might well have been influenced by quench- and strain-ageing taking place during the test. The specimens were

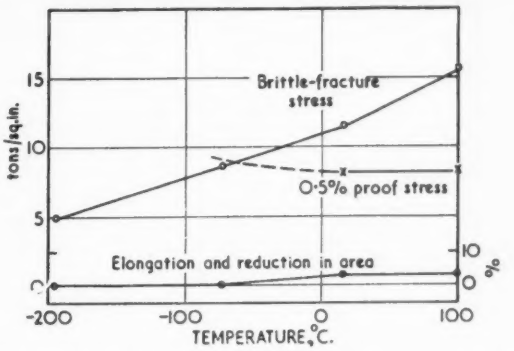

Fig. 4—Effect of temperature on tensile properties of alloy 44*AF*2 after being F.C. from 1150° C. and W.Q. from 600° C.

Table V

BRITTLE-FRACTURE STRESSES AT — 196° C. OF ALLOY 44*AF*2

After furnace-cooling from 1150° C., heating at 600° C. for the stated periods, and water-quenching.

Time of Heating at 600° C.	Brittle-Fracture Stress at — 196° C., tons/sq. in.
4 hr. 36 min.	4·9
1 hr. 10 min.	5·5
17 min.	5·5
4 min.	5·8
1 min.	5·8

completely brittle at — 196° and — 73° C., but at room temperature and 100° C. they were beginning to show slight signs of ductility as indicated by the reduction in area figures of 3%. Proof stresses were thus obtained at these higher testing temperatures. The fractures were almost entirely intergranular with only occasional cleavage facets in all the specimens.

The change in brittle-fracture stress with increase in testing temperature was thus not large, and the severe brittleness found in tension at — 196° C. after water-quenching from the range 600–700° C. showed little improvement at room temperature. There seem to be no previous determinations of brittle-fracture stress over a comparably wide range of temperature.

MICROSTRUCTURE

The microstructures of the alloys furnace-cooled, air-cooled, and water-quenched from 950° C. are shown in Figs. 5*a–c*. In the furnace-cooled condition the ferrite matrix contained needles (probably plates) which were oriented in three directions in any one grain and hence lay along (100) planes. This is confirmed by the cleavage facet in the section shown in Fig. 6 being parallel with one batch of needles; the cleavage facets were shown by back-reflection Laue photographs to be (100) planes. Furnace-cooling from 1150° C. gave a similar microstructure. This precipitate has been seen by Dijkstra[4] and by Josefsson and Kula.[11] Dijkstra obtained this nitride by heating a water-quenched alloy at 250° C., whereas Josefsson and Kula found it in furnace-cooled nitrided commercial iron. Jack[12] has determined the structure of a nitride $Fe_{16}N_2$ obtained in a similar way to that of Dijkstra, and it is now assumed that this is the precipitate which appears on (100) planes. This nitride has a distinct tendency to appear along sub-grain boundaries, but the frequency of its presence at main grain boundaries is no more than would be expected from a random distribution. Where a sub-grain boundary is approximately parallel with a (100) plane, a continuous film of nitride is obtained, and where the sub-boundary departs from this direction the film splits up into short closely spaced needles arranged *en echelon*. The nitride Fe_4N was shown by Mehl, Barrett, and Jerabek[13] to precipitate along (210) planes, giving twelve possible directions on a section. Dijkstra and Jack obtained Fe_4N by prolonged tempering of a water-quenched alloy at 250° C., but Josefsson and Kula obtained some Fe_4N with $Fe_{16}N_2$ in their furnace-cooled material. Occasional Fe_4N needles were seen in the furnace-cooled alloys in this work, but to a considerably smaller extent than found by Josefsson and Kula. Since the furnace-cooling rate in this work was very slow, 4 days being taken to cool from 950° C. to room temperature, it seems that this difference could be accounted for not by cooling-rate differences but rather by the higher nitrogen content of 0·022% of the material used by Josefsson and Kula compared with 0·01% in this work. The Fe_4N, as well as appearing in more than three directions in a grain, can be distinguished from $Fe_{16}N_2$ because the needles are usually much larger, several needles often meet at a point, and their ends tend to be pointed, whereas the $Fe_{16}N_2$ is more rod-like in appearance.

In the material air-cooled from 950° C. shown in Fig. 5*b*, a number of the grains had a mottled appearance characteristic of an unresolved precipitate. After water-quenching from 950° C., the structure consisted of clear polygonal grains, as shown in Fig. 5*c*. A similar structure was obtained by water-quenching from 300° C., at which temperature the nitrogen was all in solution.

Jack and Dijkstra have shown that $Fe_{16}N_2$ forms at an intermediate stage in the tempering at 250° C. of water-quenched material, and Fe_4N forms after prolonged tempering. That $Fe_{16}N_2$ is not stable at 200–250° C. has been confirmed in this work by heating one of the alloys at 200–250° C. after furnace-cooling from 950° or 1150° C. The $Fe_{16}N_2$ needles initially present disappeared with time and were replaced by Fe_4N needles. Figure 7 shows a few Fe_4N needles after heating for 500 hr. at 250° C. Ghosts of the $Fe_{16}N_2$ needles present originally in this specimen were also seen on the etched section, but they are difficult to reproduce in a photograph. After heating at 200° C. many more needles of Fe_4N were obtained because of the lower solubility at this temperature. From this microscopic work it appears that the 0·01% of nitrogen in these alloys starts precipitating at some temperature between 250° and 300° C.

DISCUSSION

The minimum brittle-fracture stress in the iron–0·01% nitrogen alloys dealt with in this paper was obtained after heating in a temperature range about 300–400° C. above the temperature at which all the nitride goes into solution. Consequently, an explanation of this effect has been suggested in terms of segregation of nitrogen at grain boundaries without the separation of a second phase. But it is necessary to consider why higher fracture stresses are obtained after heating at temperatures both above and below 600–700° C. The improvement in the strength of the grain boundaries after heating above 700° C. could well be due to thermal vibrations dispersing the concentration of nitrogen at the boundaries and giving a nearer approach to a uniform distribution of the nitrogen atoms in the solid solution. The reason for the increase in strength of the grain boundaries after heating below 600° C. but still within the single-phase region could be that the grain-boundary segregation is diminished by an association of iron and nitrogen atoms within the grains. Such a non-uniform distribution of nitrogen atoms might be expected as a preliminary stage to precipitation of nitride. If the association postulated here were the forerunner of precipitation of $Fe_{16}N_2$ which appears on slow cooling below 300° C., the rate of increase of fracture stress might be expected to be greatest just above the temperature at which precipitation starts. However, the results in Fig. 3 show that there was a very sharp improvement in fracture stress on lowering the temperature of treatment from 500° to 400° C., whilst there was little additional improvement on further lowering the temperature of heating to 300° C. If the strengthening of the grain boundaries after heating below 600° C. is caused by forces that encourage

(a) (b) (c)

Fig. 5—Alloy 44*AF*1: (*a*) F.C.; (*b*) A.C.; and (*c*) W.Q. from 950° C., showing (*a*) $Fe_{16}N_2$; (*b*) an irresolvable precipitate in some grains; and (*c*) clear polygonal grains × 500

Fig. 6—Section through cleavage facet in a brittle specimen of alloy 44*AF*1 F.C. from 950° C., showing $Fe_{16}N_2$ needles parallel to the facet × 1500

Fig. 7—Alloy 44*AF*1 F.C. from 950° C. and heated for 500 hr. at 250° C., showing needles of Fe_4N and ghosts of $Fe_{16}N_2$ × 500

clustering of nitrogen atoms within the grains, there is some indication that this clustering or association may not be precisely that expected to precede precipitation of $Fe_{16}N_2$.

CONCLUSIONS

The addition of about 0·01% of nitrogen to high-purity iron caused the appearance of intergranular brittleness that was sensitive to heat-treatment. The proportion of intergranular failure was larger as the cooling rate from the austenitic range increased and was accompanied by a higher transition temperature in impact and a lower brittle-fracture stress in tension at — 196° C. Ageing the air-cooled or water-quenched material at room temperature caused a small lowering of the impact transition temperature which could be ascribed to a small rise in brittle-fracture stress. Furnace-cooling from 950° to 300° C. and water-quenching to retain the nitrogen in solution gave a somewhat lower impact transition temperature than furnace-cooling to room temperature, probably because of a small superiority in brittle-fracture stress.

It has been shown by first obtaining the material in its toughest condition by furnace-cooling, and then applying isothermal treatments at 100° intervals from 300° to 1000° C. and water-quenching, that maximum embrittlement of the grain boundaries occurred at 600–700° C. The remarkably low brittle-fracture stress at — 196° C. of about 5 tons/sq. in. was obtained by heating at 600–700° C. followed by water-quenching, and this was consistent with the large proportion of intergranular failure in the fractures. The time required at 600° C. to produce maximum embrittlement was very short, the shortest time used (about 1 min.) being sufficient. The simplest explanation of the results at present is that there is a maximum tendency for nitrogen to diffuse to the grain boundaries at 600–700° C., whereas above and below this temperature range the tendency to diffuse away from the grain boundaries becomes progressively stronger.

After heat-treatment to give the most brittle condition, the brittle-fracture stress increased linearly with temperature from — 196° C. to room temperature; tests at 100° C. showed that the rise in fracture stress was somewhat more rapid above room temperature but that the material was still almost completely brittle in tension at 100° C.

The nitride $Fe_{16}N_2$ was precipitated in these alloys by furnace-cooling to room temperature, although occasional needles of Fe_4N were also seen. It has been confirmed that heating at 200° or 250° C. for prolonged periods results in the disappearance of $Fe_{16}N_2$ and the formation of Fe_4N.

Acknowledgments

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Pearlite-Free Basic Bessemer Steel: Its Fabrication and Properties

By Åke Josefsson

SYNOPSIS

Steels containing less than 0·015% of carbon, here termed 'pearlite-free', are characterized by much lower impact transition temperatures than steels with similar base composition and slightly higher carbon contents. Provided that this carbon limit is not exceeded, phosphorus and nitrogen up to 0·15% and 0·020% respectively may be used as alloying additions to give a yield strength comparable with that of plain carbon steel containing 0·15–0·20% C, without noticeable impairment of the impact properties even after straining and ageing. The manganese content should be kept rather low, about 0·20%; higher percentages are probably unfavourable to low-temperature ductility after slow cooling. Pearlite-free steels of this type are shown to be easily and economically produced by the basic Bessemer process. Examples of results from impact tests on plates of these steels are given, together with results of bend tests on welded specimens. Corresponding data for plain carbon steels are included for comparison. 880

Introduction

As a result of, *inter alia*, the increased use of welding for joining heavy steel sections, there has arisen a growing demand for good low-temperature ductility in ordinary mild steels for constructional purposes. This has been rather awkward, especially for makers of basic Bessemer steels, as such steels in many cases have proved insufficient in this respect. Their inferiority has often been traced back to the higher percentages of phosphorus and nitrogen, by which they generally differ from open-hearth steels. The unfavourable influence of these two elements upon ductility has been proved in many investigations, and this, in turn, has stimulated the development of special blowing methods, particularly using oxygen, by which basic Bessemer steels may be produced that are comparatively low both in phosphorus and nitrogen. It is remarkable, however, that little attention has been paid to the influence of carbon, although nitrogen and carbon seem to be very similar in regard to their behaviour in the alpha-iron structure. One reason for this negligence may be the conventional conception that carbon is indispensable as a strengthening and hardening constituent in the steel; another may be the fact that no noticeable effect on ductility appears until the carbon content has been reduced to about 0·015%, a value which is never (or at least very seldom) obtainable in ordinary steelmaking processes. With the aid of extended refining *in vacuo*, however, it has been possible to produce steels with these low carbon contents on a laboratory scale, and to investigate the influence of carbon on the low-temperature ductility in this carbon range. Some years ago such investigations were carried out at Domnarfvet, the 'transition temperature' in impact testing of Charpy specimens being used as a criterion. It appeared that the transition temperature decreased very markedly when the carbon was reduced to below 0·015%, manganese, sulphur, phosphorus, and nitrogen being present in amounts normally met with in ordinary mild open-hearth steels. Moreover, it was proved that if the carbon content was less than 0·015% the transition temperature remained low even though considerable amounts of phosphorus and nitrogen were added. This discovery led to the development of a weldable, low-carbon basic Bessemer steel[1] which is to be described in this paper.

IMPORTANCE OF A LOW CARBON CONTENT FOR LOW-TEMPERATURE DUCTILITY

The discontinuous decrease in transition temperature occurring when the carbon content is reduced from 0·02 to 0·01% is very clearly demonstrated, in the case of pure Fe–C alloys, by Allen *et al.* in a recently published paper.[2] When other impurities are present in amounts normal for commercial steels, this reduction in carbon content also causes a decrease in transition temperature which is of the same order of magnitude. This is shown in Fig. 1, in which impact-energy/temperature curves are given for the two groups of experimental steels with carbon contents of 0·01% and 0·02%. These steels were melted in a high-frequency vacuum furnace (capacity 15 kg.) as far as possible maintaining identical conditions, using the same low-carbon melting stock, and graphite, ferro-phosphorus, and nitrided iron powder as alloying additions. For comparison, a steel with higher carbon and manganese contents is also included (*K*40); this was melted in the same way and is intended to represent a plain carbon steel. The ingots were forged to 15-mm. square rods, normalized for 15 min. at 950° C., and tested in impact* over a range

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* Unless otherwise stated, all impact tests reported in this paper refer to standard-size Charpy bar with keyhole notch and broken by a pendulum with capacity 15 kg.m.

of temperatures, both normalized only (Fig. 1*a*) and normalized, strained 10%, and aged at 200° C. for 6 hr. (Fig. 1*b*). The analyses, yield-point values, and grain-size numbers (JKM scale) are given in Table I.

Figure 1 shows that the steels with carbon contents of about 0·01% have their transition curves shifted 50–80° C. towards lower temperatures, compared with the two steels containing 0·02% C. This shift is somewhat larger in the non-aged condition than after straining and ageing. Heat *K*40 (representing the conventional carbon steels), although very low in phosphorus and nitrogen, has its transition curves in the same range as the two steels with 0·02% C. Consequently, it seems that there is very little to be gained, in respect of transition temperature, from thorough removal of phosphorus and nitrogen in plain carbon steels, compared with the effect of reducing carbon to about 0·01%. This is valid even when phosphorus and nitrogen are reduced below the present practical limit.

The critical carbon content below which the shift in transition temperature seems to be completed has been found to be about 0·015%. This value is not very far from the limit below which no pearlite appears in the microstructure of an air-cooled specimen; therefore, the term ' pearlite-free ' may be used for this type of steel.

For most constructional steels, however, the load-carrying capacity is of primary importance. This property is most adequately described by the yield-strength value σ_y, which is given in Table I for all the heats quoted above. According to Table I, a low-carbon, pearlite-free steel with 0·066% P and 0·016% N has a yield strength of 26·7 kg./sq. mm., which is considerably higher than that of the ' standard ' steel *K*40. The general conclusion from these tests, therefore, is that the attainment of a yield strength of 24–27 kg./sq. mm. in a steel by increasing the phosphorus and nitrogen contents is less damaging to the low-temperature ductility than if this yield strength is obtained by additions of carbon and manganese.

Rôle of Carbon in Brittle Fracture

The shift in transition temperature effected by reducing the carbon content below 0·015% may be compared with that obtained by quenching a previously furnace-cooled steel containing about 0·015% C. Allen and his co-workers[2] considered that, in both cases, the disappearance of the grain-boundary

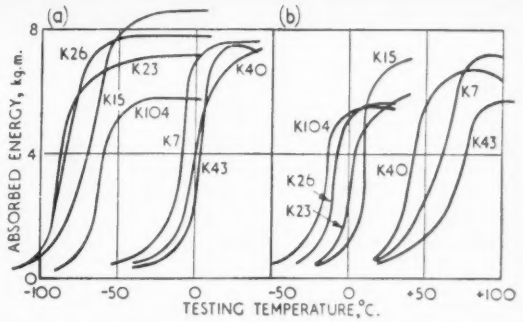


Fig. 1—Impact transition curves for H.F. experimental heats of various steels. (*a*) Normalized only; (*b*) normalized, strained, and aged

cementite was the immediate cause of the change. This constituent is said to induce brittle fracture by being cracked at an early stage of deformation in the impact test, thus introducing internal notches in the steel. In order to illustrate further the rôle of carbon in brittle fracture, some experiments on low-carbon and carbon-free steels of commercial purity will be reported.

Bars 15 mm. square were rolled from a low-carbon, aluminium-stabilized steel *A*, the composition of which is given in Table II. Specimens were slowly cooled from 950° C. to the respective temperatures of 650°, 600°, 550°, and 500° C. and quenched in brine. After ageing at 100° C. for 3 days and an additional strain-ageing (straining 10%, ageing at 200° C. for 6 hr.), impact testing at different temperatures was carried out. The results (Fig. 2*a*) show that transition temperatures shift considerably upwards when slow cooling is extended to temperatures lower than 600° C. Grain-boundary cementite could be detected in the microstructure of the specimens slowly cooled to 550° and 500° C. but not in the other two specimens.

Bars of this steel were also decarburized in a stream of hydrogen (saturated with water at 18° C.) at a temperature of 700° C. After 13 days the carbon content was found to be less than 0·001%. One bar (*A*1) was tested directly after this treatment, and another after heating to 950° C., air-cooling, reheating to 700° C., and quenching; both treatments were carried out in an argon atmosphere. Testing consisted of strain-ageing and impact testing as described previously. In both cases the transition temperature

Table I

COMPOSITION, YIELD STRENGTH, AND GRAIN SIZE FOR VACUUM-MELTED 15-kg. HEATS

Heat No.	Analysis					Yield Strength, σ_y kg./sq. mm.	Grain Size, JKM No.
	C, %	Mn, %	P, %	S, %	N, %		
K15	0·011	0·24	0·002	0·019	0·003	16·0	11½
K23	0·009	0·26	0·046	0·022	0·003	23·2	10½
K26	0·007	0·25	0·061	0·025	0·003	22·3	10½
K104	0·007	0·25	0·066	0·016	0·016	26·7	11½
K7	0·020	0·26	0·011	0·017	0·002	20·2	11
K43	0·019	0·24	0·015	0·008	0·015	28·2	10
K40	0·15	0·46	0·003	0·007	0·005	24·0	10

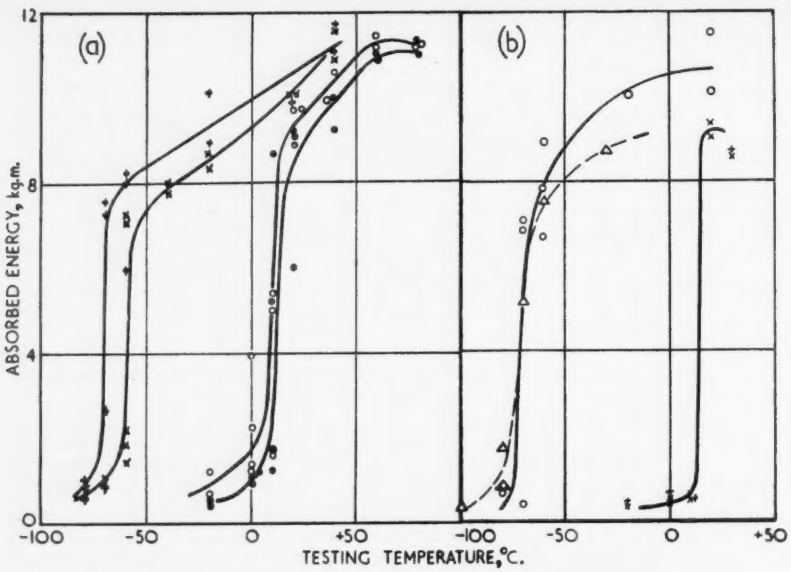

(a)
(b)
ABSORBED ENERGY, kg.m.
TESTING TEMPERATURE, °C.
× F.C. 950–650° C.
+ F.C. 950–600° C.
○ F.C. 950–550° C.
● F.C. 950–500° C.
After brine-quenching, specimens aged 3 days at 100° C., strained 10%, and aged 6 hr. at 200° C.
○ F.C. 950–700° C., brine-quenched, aged 4 days at 100° C., strain-aged
× Decarburized to < 0·001% C, strain-aged
+ Decarburized to < 0·001% C, normalized at 950° C., reheated to 700° C., brine-quenched, strain-aged
Δ Decarburized to < 0·001% C, recarburized to 0·005% C, reheated to 700° C., brine-quenched, strain-aged

Fig. 2—Impact transition curves for aluminium-stabilized low-carbon steel *A*: (*a*) After quenching from different temperatures; (*b*) after decarburizing and recarburizing

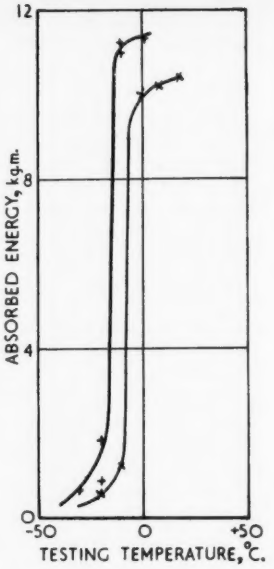


Fig. 3—Impact transition curves for titanium-stabilized low-carbon steel *B*

is about 20° C. (*see* Fig. 2*b*). It may be emphasized that no trace of intercrystalline cracks could be found by microscopical examination; all fractures were entirely of the cleavage type.

A third bar (*A*2) was recarburized to 0·005% C by heating to 650° C. for 7 days in hydrogen that had been saturated with *n*-heptane at room temperature. Impact testing after reheating for 30 min. at 700° C., quenching, straining, and ageing as previously, gave a transition curve almost identical with that of the original material (prior to decarburization) after the same heat-treatment (Fig. 2*b*).

Carbon elimination may also be effected by adding an amount of titanium sufficient for the fixation of all carbon as titanium carbide, as in steel *B*, the base composition of which is about the same as that of steel *A* (*see* Table II). Steel *B* was melted *in vacuo* in a high-frequency furnace and forged to 15-mm. square bars; specimens were tested in impact after quenching or furnace-cooling from 700° C., straining, and ageing. The results are shown in Fig. 3. In neither case was any grain-boundary constituent observed in the microstructure.

The results may be interpreted as follows. The transition temperature of steel *A*, heat-treated so that it contained grain-boundary carbide, is almost the same as that of a ferrite with the same base composition but essentially free from both grain-boundary carbide and soluble carbon, irrespective of cooling rate in the α range. On the other hand, the presence of a certain amount of soluble carbon makes it possible to attain very much lower transition temperatures, provided that the steel is quenched from a temperature around the A_1 point. This, in turn, seems to indicate that the transition temperature is not affected by the presence of grain-boundary carbide *per se*, but is mainly determined by the properties of the ferrite, a ferrite with some finely dispersed

Table II

COMPOSITIONS OF EXPERIMENTAL STEELS WITH ALUMINIUM AND ALUMINIUM–TITANIUM ADDITIONS

Notation	C, %	Mn, %	P, %	S, %	N, %	Al (sol.), %	Ti, %
A	0·010	0·13	0·026	0·017	0·026	0·065	...
A1	<0·001	...	...	...	0·026	...	...
A2	0·005	...	...	...	...	...	...
B	0·013	0·15	0·009	0·022	0·007	0·066	0·27

carbide having a much lower transition temperature than a ferrite in which this phase is missing. The presence of grain-boundary carbide should then be regarded merely as an indication of the absence of this fine dispersion.

Possible mechanisms for the phenomena described are discussed elsewhere.[3]

In the same way, the large shift in impact temperature of air-cooled steel accompanying a reduction of carbon content to 0·015% may be due to the occurrence of carbon as a finely dispersed precipitate, rather than to the simultaneous disappearance of grain-boundary cementite. The condition that carbon should be lower than 0·015% may then form the prerequisite for the spontaneous formation of such a precipitate during air-cooling.

PRINCIPLES FOR PRODUCTION OF PEARLITE-FREE HIGH-PHOSPHORUS STEELS IN THE BASIC-BESSEMER CONVERTER

It would be almost impossible, or at least very expensive, to make a steel with less than 0·015% C on a tonnage scale by any known technical process except by Bessemerizing. With regard to carbon removal, the Bessemer process bears some resemblance to melting *in vacuo* but has not the disadvantage of the high costs of the latter. This paper will deal only with the basic Bessemer process, by which steels with less than 0·015% C and with nitrogen and phosphorus in appropriate amounts may be produced fairly easily.

The characteristic feature of a normal basic Bessemer blowing (outlined in Fig. 4) is that the carbon is reduced to rather low percentages while the phosphorus content is still almost unchanged; the phosphorus oxidation starts essentially only after the formation of a fluid slag with high basicity, capable of binding phosphorus pentoxide. During the period of phosphorus refining, some further reduction of the carbon takes place. The final carbon content after completed blowing is generally accepted as 0·02–0·03%, but values both above and below this range may be found in the literature; disagreements may be due to the lack of accuracy in the methods of analysis applied earlier, more than to true differences. Using modern and more exact methods for carbon determination at low percentages, it has been possible to show that a further reduction of the final carbon content to 0·01% and below is possible, provided that the process is conducted so as to sufficiently delay the phosphorus reaction. A low temperature at the end of the carbon boil is beneficial for this purpose. If such a blow is interrupted at 0·100% P, the composition of the melt will meet the requirements for a pearlite-free steel as outlined in the foregoing paragraph. It can be poured into ingots directly or with additions for deoxidation and increasing manganese content, if desired. The liquid steel must not, of course, come into contact with any carbonaceous matter, *e.g.*, tarred dolomite or mould-dressing containing graphite or pitch, as carbon is taken up rather eagerly by the steel. Ferro-alloys, if added, would have to be sufficiently low in carbon.

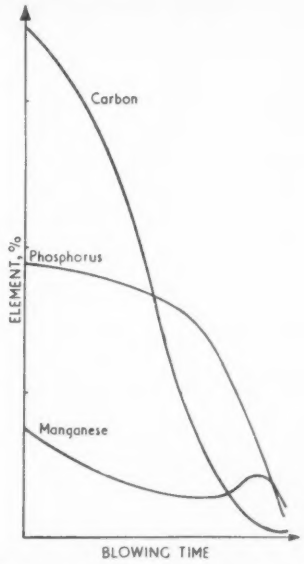


Fig. 4—Carbon, phosphorus, and manganese contents in steel bath during basic Bessemer blowing (schematic)

An important point is that if the blowing is carried out correctly the oxygen content at, say, 0·100% P will not be higher than about 0·03%, corresponding to a carbon content of 0·06% if the carbon–oxygen equilibrium relation is applied. Thus there is generally no need for large amounts of deoxidants to be added; even with no additions the pearlite-free steel solidifies in the same way as an ordinary semi-killed steel. The reason for this is that, during the final period of blowing, the oxygen content is governed by the equilibrium between phosphorus, oxygen, and slag, which prescribes very much lower oxygen activity in the metal than the carbon reaction does, owing to the high affinity between phosphorus pentoxide and a lime-rich slag in the actual temperature range. One manifestation of these relations is the manganese reversion, indicating a reduction of manganese from the slag into the steel bath. This causes the well-known ' hill ' on the manganese curve in Fig. 4, which marks the highest rate of phosphorus oxidation. The intense reduction of carbon, despite a low and even decreasing oxygen content, is due to the fact that the carbon monoxide pressure cannot be assumed to be equal to 1 atm. (as, for instance, in refining in open-hearth or electric-arc furnaces) but is continuously kept to very low values, since the carbon monoxide formed is immediately swept away from the gas–metal surface by the big quantities of other gases (mainly nitrogen) which are continually bubbling through (or passing the surface of) the molten steel.

In most ordinary basic Bessemer steels very low phosphorus contents are desired—certainly not more than 0·050%. The additional blowing required for this generally leads to a considerable increase in the oxygen content in the steel, and is also reflected in a further decrease in manganese content in the metal and a rapidly rising iron oxide content in the slag.

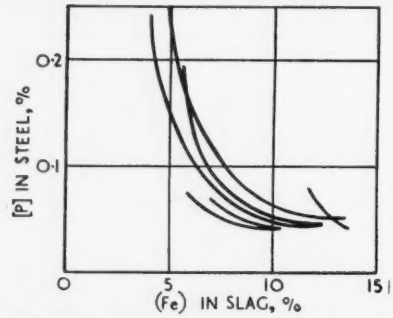


Fig. 5—Phosphorus content in steel bath plotted against iron content in slag in basic Bessemer process (from Geller and Wilms[4]). The curves represent results from different investigators

Figure 5 shows the iron oxidation as a function of the final phosphorus content.

A low manganese content in the steel makes it necessary to watch the sulphur content, otherwise the process of hot-rolling will be troublesome. At 0·20% Mn, however, no serious difficulties will be met until the sulphur content exceeds 0·030%, provided that the steel is heated to a sufficiently high temperature (1150–1200° C:). The first passes in the blooming mill seem to be critical. Rolling slabs or billets does not cause cracks, even at still higher sulphur contents and much lower temperatures.

The process described would seem to make practical use of the good ductility properties of the pearlite-free steels and to widen the field of application of the basic Bessemer process. However, this would be done not by reducing the phosphorus and nitrogen contents as low as possible, as in present practice, but by making the steel immune from the embrittling action of these elements, which may then freely be used for securing a higher strength in the steel.

Effect of Manganese

Occasionally some preliminary full-scale heats of pearlite-free basic Bessemer steel, especially when slowly cooled, showed unexpectedly high transition temperatures after strain-ageing. It was established that this behaviour was in some way connected with the manganese content; it could appear at about 0·30–0·40% Mn, but was never found at or below

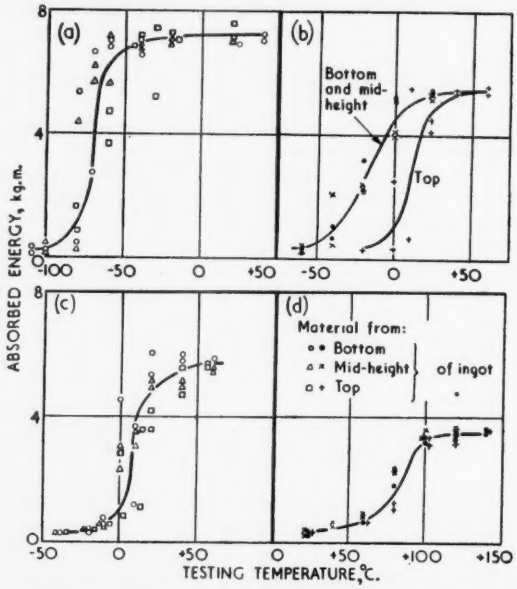


Fig. 6—Impact transition curves for pearlite-free basic Bessemer steel 74: (*a*) Normalized; (*b*) normalized, strained, and aged; (*c*) furnace-cooled; (*d*) furnace-cooled, strained, and aged

0·20% Mn. The mechanism of this effect is not clear at present. The inference is, however, that when the steel is to be used for plates or structurals in heavier gauges the manganese content should be limited to 0·20%, unless arrangements are made for accelerated cooling after hot rolling.

PROPERTIES OF PEARLITE-FREE BASIC BESSEMER STEEL

Tensile and Impact Properties

The following survey was made in order to evaluate the degree of uniformity in a full-sized ingot of pearlite-free basic Bessemer steel, but it is also intended to give a comparison between the mechanical properties of this steel and two plain carbon steels, particularly in regard to low-temperature ductility.

In a basic-lined converter with a capacity of 14 tons, a heat of pearlite-free steel (74) was made by

Table III

COMPOSITIONS OF A PEARLITE-FREE STEEL AND OF TWO ORDINARY STEELS

Steel	Position in Ingot	Chips From Section					Borings From Centre	
		C, %	Mn, %	P, %	S, %	N, %	C, %	P, %
Pearlite-free basic Bessemer (steel 74)	Bottom	0·012	0·40	0·087	0·026	0·015	0·010	0·081
	Mid-height	0·011	0·40	0·090	0·026	0·015	0·011	0·085
	Top	0·012	0·40	0·086	0·026	0·015	0·011	0·110
Rimmed basic Bessemer (steel 72)	Bottom	0·06	0·45	0·018	0·018	0·013	0·05	0·015
	¼-height	0·07	0·47	0·030	0·024	0·013	0·10	0·049
	Mid-height	0·07	0·47	0·034	0·024	0·013	0·09	0·034
Killed electric-arc furnace (steel 27) (Si = 0·20%)	Bottom	0·13	0·45	0·008	0·022	0·005	...	...

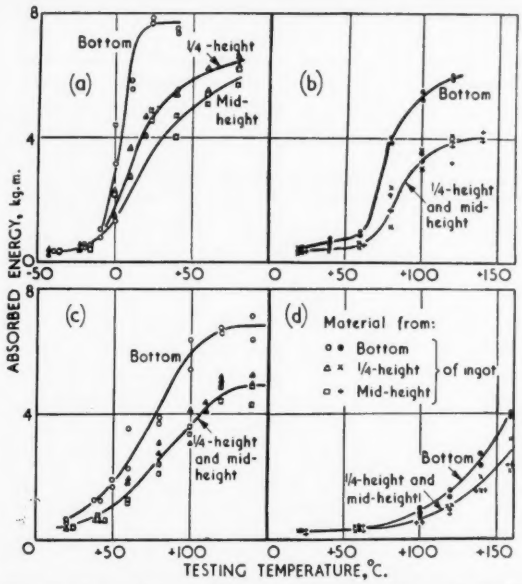


Fig. 7—Impact transition curves for rimmed basic Bessemer steel 72: (*a*) Normalized; (*b*) normalized, strained, and aged; (*c*) furnace-cooled; (*d*) furnace-cooled, strained, and aged

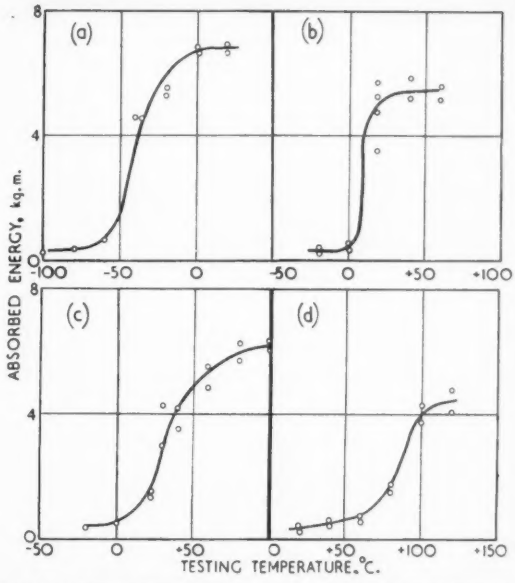


Fig. 8—Impact transition curves for electric-arc furnace steel 27, Si-killed: (*a*) Normalized; (*b*) normalized, strained, and aged; (*c*) furnace-cooled; (*d*) furnace-cooled, strained, and aged

blowing down to about 0·080% P and adding 3 kg. of electrolytic manganese per ton of steel in the ladle. The steel was poured into ordinary 3-ton ingots, which were rolled to 100-mm. square billets. Billets corresponding to different positions in the ingot were selected for investigation, together with similar billets rolled from 3-ton ingots of an ordinary rimming basic Bessemer steel (72) and an ordinary silicon-killed electric-arc furnace steel (27), for comparison. Chips milled from sections of these billets were analysed. For the two basic Bessemer steels, carbon and phosphorus analyses were also made on borings from the centres of the sections.

The results, given in Table III, show that the differences in composition due to segregation are much bigger, proportionately, in the rimmed steel 72 than in the pearlite-free steel 74, even though the top billet of steel 72 is not taken into consideration.

The billets were further rolled to bars 15 mm. in dia. (steels 74 and 27) and 15 mm. square (steel 72). After normalizing or furnace-cooling from 950° C., tensile testing gave the results reported in Table IV. The tensile properties of the pearlite-free steel 74 are fairly uniform along the whole ingot, whereas for steel 72 appreciable differences appear even between billets from bottom and mid-height of the ingot. Moreover, steel 74 has throughout a higher yield strength than steels 72 and 27, in both the normalized and the furnace-cooled conditions.

Impact tests at different temperatures have been

Table IV

TENSILE PROPERTIES CORRESPONDING TO ANALYSES QUOTED IN TABLE III

Each value is a mean of two tests

Steel	Position in Ingot	Normalized From 950° C.				Furnace-Cooled From 950° C.			
		Yield Strength, σ_y kg./sq. mm.	Ultimate Strength, σ_u kg./sq. mm.	Elongation, δ_{10} %	Red. in Area, ψ %	Yield Strength, σ_y kg./sq. mm.	Ultimate Strength, σ_u kg./sq. mm.	Elongation, δ_{10} %	Red. in Area, ψ %
Pearlite-free basic Bessemer (steel 74)	Bottom	32·6	43·4	26	72	28·1	40·6	29	73
	Mid-height	33·0	44·0	27	72	27·0	41·3	29	74
	Top	32·9	44·3	26	72	27·3	41·6	28	73
Rimmed basic Bessemer (steel 72)	Bottom	25·6	38·7	31	75	20·4	35·4	32	76
	¼-height	27·0	41·9	29	70	22·3	38·3	31	70
	Mid-height	28·8	44·0	28	69	23·8	39·8	29	68
Killed electric-arc furnace (steel 27)	Bottom	27·0	40·9	29	65	23·2*	40·6*	30*	64*

* These values were obtained on another steel with almost the same analysis as steel 27

Table V

CHANGES IN TENSILE PROPERTIES DURING AGEING

Steel	Preliminary Heat-Treatment	Ageing Treatment		Tensile Properties		
		Temp., °C.	Time	Yield Strength, σ_y kg./sq. mm.	Ultimate Strength, σ_u kg./sq. mm.	Elongation, δ_{10} %
Pearlite-free basic Bessemer (steel 74)	Normalized at 950° C.	20	...	29·6	...	...
		,,	4 weeks	31·4	43·5	28·0
		,,	8 ,,	32·7	44·4	26·3
		,,	17 ,,	34·7	44·3	24·5
		,,	68 ,,	35·5	42·8	26·7
		,,	4½ years, approx.	33·7	...	...
		50	...	32·8	44·0	25·8
		,,	3 days	35·5	44·2	21·7
		,,	2 weeks	34·6	41·8	23·9
		,,	6 ,,	34·6	42·0	24·2
		,,	12 ,,	35·1	41·8	22·2
		,,	26 ,,	34·1	41·5	25·4
		200	24 hr.	28·0	39·4	31·4
		,,	3 days	26·2	38·4	31·6
		,,	7 ,,	25·3	37·3	32·2
		,,	28 ,,	25·8	37·2	33·4
	Furnace-cooled from 950° C.	20	...	25·8	...	...
		,,	2 weeks	26·1	...	...
		,,	20 ,,	27·0	...	...
		,,	78 ,,	28·2	...	...
Semi-killed electric-arc furnace (steel 27) (C = 0·10%, Si = 0·12%, Mn = 0·70%)	Normalized at 870° C.	200	...	30·1	42·8	29·5
		,,	24 hr.	29·5	42·6	30·2
		,,	3 days	29·5	42·6	32·1
		,,	28 ,,	26·6	41·7	30·1

made on the steels directly after heat-treatment, as well as after additional strain-ageing (straining 10%, ageing for 6 hr. at 200° C.). The resulting transition curves are shown in Figs. 6, 7, and 8. Even here the divergences of the results for different parts of the ingot are rather small for the pearlite-free steel—only in one case (after normalizing and strain-ageing) has a separate transition curve had to be drawn for the top material (Fig. 6*b*). The impact results for rimmed steel 72, on the other hand, are considerably different even for the bottom and mid-height positions. On the whole, it may be stated that the transition curves of the pearlite-free steel lie in the same range as those of killed steel 27, whereas the curves of rimmed steel 72 are shifted substantially towards higher temperatures. This difference between pearlite-free and ordinary basic Bessemer steels is more pronounced after normalizing than after slow furnace cooling.

Effect of Ageing

A comparatively high nitrogen content may be expected to render the pearlite-free steels prone to ageing, *i.e.*, disposed to change their properties when stored for a long time at atmospheric or moderately elevated temperatures. To find the extent of these changes and how they affected the impact properties, bars from pearlite-free steel 74 (*see* Tables III and IV) were tested after normalizing—a few also after furnace-cooling—and subsequent ageing, as follows:

(i) Tensile tests after ageing at room temperature, 50° C., 100° C., and 200° C. for different periods

Table VI

CHANGES IN TRANSITION TEMPERATURE DURING AGEING OF PEARLITE-FREE BASIC BESSEMER STEEL

Heat-Treatment	Ageing Treatment		Transition Temperature, °C.	
	Temp., °C.	Time	Unstrained	Strain-Aged
Normalized	...	...	−70	−20
	20	20 weeks	−40	−10
	,,	18 months	−40	−15
	,,	54 ,,	−55	−15
	50	3 days	−60	−20
	,,	6 weeks	−50	−20
	,,	12 ,,	−40	−25
	,,	16 ,,	−40	−25
	,,	20 ,,	−60	−20
	,,	26 ,,	−55	−25
	200	24 hr.	−35	10
	,,	3 days	−20	15
	,,	7 ,,	−20	5
	,,	28 ,,	−20	0
Furnace-cooled	...	...	10	90
	20	2 weeks	5	80
	,,	20 ,,	0	70
	,,	18 months		70

Table VII

STEEL AND SLAG ANALYSES FOR PEARLITE-FREE BASIC BESSEMER STEELS

Heat	Steel						Slag	
	C, %	Si, %	Mn, %	P, %	S, %	N, %	Fe, %	Mn, %
PF–1	0·005	0·07	0·21	0·093	0·035	0·015	8·6	3·0
PF–2	0·008	0·09	0·20	0·093	0·032	0·015	6·8	2·5
PF–3	0·008	0·03	0·22	0·100	0·031	0·012	5·4	2·5
PF–4	0·007	0·02	0·20	0·100	0·027	0·016	6·6	1·8
R1	0·07	...	0·40	0·068	0·036	0·013		
FK	0·17	0·25	0·80	0·013	0·026	0·007		

(ii) Impact tests over a range of temperatures after ageing at room temperature, 50° C., and 200° C. for different periods. Testing was carried out directly after these ageing treatments, as well as after subsequent strain-ageing

(iii) Impact tests at various temperatures after straining 10% and ageing at room temperature, 100° C., and 200° C. for different periods.

Tests (iii) were carried out to establish whether strain-ageing embrittlement could be developed to a higher degree by selecting some other temperature and time for the artificial ageing than the usual 200° C. at 6 hr.

The following results were obtained:

Tests (i)—The tensile values (Table V) show for the normalized condition a moderate upward trend for the yield strength, during the first few weeks of ageing at room temperature, up to a value of 34–35 kg./sq. mm., which then seems to be retained even for long ageing times. When the ageing temperature is raised to 50° C. the same yield strength is reached earlier and is then retained at the same level. At 200° C., over-ageing occurs almost immediately and the yield strength decreases by about 2 kg./sq. mm.; this also occurs in the ordinary carbon steel 27. In the furnace-cooled condition, also, the pearlite-free steel experiences a minor rise in yield strength when aged at room temperature.

The variations in yield strength are to some extent reflected also in the ultimate strength values, whereas the elongation varies in the opposite way.

Tests (ii)—Impact testing after the various ageing treatments gave transition curves which were similar in appearance to those in Fig. 6. However, some shifts along the temperature axis do occur. The transition temperatures after the various ageing treatments, given in Table VI, are evaluated as the temperatures at which the transition curves give the value of 2·5 kg.m. for absorbed energy. Ageing at room temperature or at 50° C., consequently, does not change the transition temperatures very much. Although some shift towards higher temperatures occurs, in the non-strain-aged condition, after 20 weeks of ageing at room temperature, a partial reversion of this rise may be noticed when ageing is further prolonged. A greater increase in transition temperature is caused by ageing at 200° C., again less pronounced in the strain-aged condition. (In fact, the transition temperature after straining and ageing shows a tendency to fall after the initial rise, when the time at 200° C. is increased.)

Tests (iii)—Ageing at room temperature, 100° C., and 200° C. for different periods following a 10% straining did not cause higher transition temperatures than those obtained by ageing at 200° C. for 6 hr. The transition temperature after straining and ageing at room temperature for 18 months, for example, proved to be — 20° C.

PROPERTIES OF PLATES MADE FROM PEARLITE-FREE BASIC BESSEMER STEEL

To show what transition temperatures and yield strengths can be expected in large-scale production, the following examples are reported.

In an ordinary, dolomite-lined converter (capacity 28 tons) a pig iron of average composition 3·4% C, 0·25% Si, 0·6% Mn, 1·7% P, and 0·06% S was blown to an estimated phosphorus content of 0·09%. After removal of the slag the steel was teemed into a ladle, with the addition of 2 kg. of ferro-silicon (45% Si) per ton, and was subsequently poured into open-top moulds. The ingots, weighing 3–4·4 tons, solidified as ordinary semi-killed steel with an almost plane upper surface (a slight convexity is believed to be more profitable), and were rolled to 15- and 20-mm. thick plates, 1500 mm. wide.

Table VII shows the composition of the steel in samples taken from the plates and corresponding to the lower parts of the ingots; the iron and manganese contents in the final converter slag before teeming are also given. The compositions of a rimmed basic Bessemer steel and a killed electric-arc furnace steel are included for comparison.

For mechanical testing, coupons were cut from both the centre and the edge of each plate. The test bars were cut from the plate in the longitudinal direction (fracture surface normal to rolling direction), but the transverse properties were also controlled to some extent. The notch was drilled with the axis normal to the plane of the rolled surface. Impact testing was carried out (*a*) directly in the as-rolled condition, (*b*) after straining 10% and ageing at 200° C. for 6 hr., and (*c*) after compressing 10% and ageing at 250° C. for ½ hr.

The tests on compressed material, which were made according to a German practice,[5] appeared to be more discriminating, for the basic Bessemer steels, than

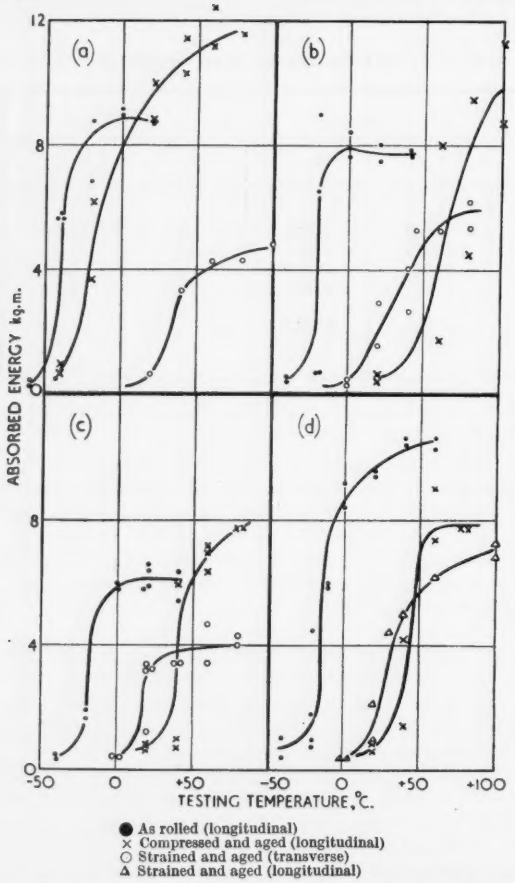


Fig. 9—Impact transition curves for pearlite-free basic Bessemer steels, as hot-rolled to 15-mm. plate. (*a*) Heat *PF*–1; (*b*) heat *PF*–2; (*c*) heat *PF*–3; (*d*) heat *PF*–4

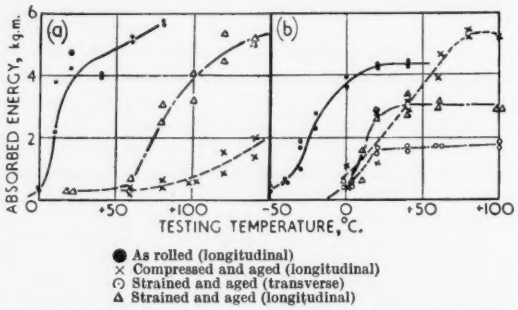


Fig. 10—Impact transition curves for basic Bessemer and killed electric-arc furnace steels, as hot-rolled to 15-mm. plate. (*a*) Heat *R*1; (*b*) heat *FK*

those in which the same deformation was made by straining. The notch on the test bar was made according to the German DVM standard, which differs slightly from the Charpy.

Some of the transition curves obtained for the 15-mm. plates are shown in Fig. 9, and the corresponding curves for the two ordinary steels are shown in Fig. 10. The results, including yield-strength values, are summarized in Table VIII. As before, the transition temperature is evaluated as the temperature at which the value of the absorbed energy is 2·5 kg.m. The accuracy is not very good, as the number of test bars broken in the critical range was in some cases insufficient and the spread was considerable; nevertheless, it is felt that Table VIII provides a fair comparison between the properties of the pearlite-free steel and the ordinary steels.

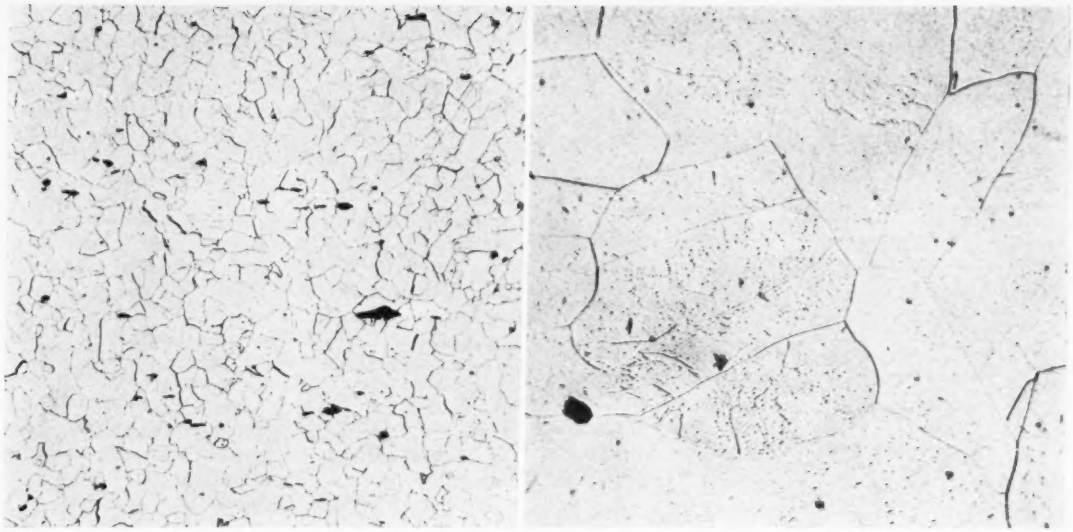
In regard to yield strength, all the steels may be considered equivalent. The transition temperatures of the pearlite-free steels agree on the whole with the fully killed steel *FK*, whereas those of the rimmed basic Bessemer steel *R*1 are considerably higher, especially in the strain-aged condition. Moreover, the energy values in the ductile range are very much

Table VIII

YIELD STRENGTHS AND TRANSITION TEMPERATURES OF PEARLITE-FREE BASIC BESSEMER STEELS

As hot-rolled to plate

Heat	Plate Thickness, mm.	Yield Strength, σ_y kg./sq. mm.		Transition Temperature, °C.					
				Longitudinal, Centre			Longitudinal, Edge	Transverse, Edge	
		Longitudinal	Transverse	As-Rolled (Charpy)	Strained and Aged (Charpy)	Compressed and Aged (DVM)	Strained and Aged (Charpy)	Strained and Aged (Charpy)	Compressed and Aged (DVM)
PF–1	15	27·3	29·9	−45	25	−20	10	35	approx. 0
	20	28·1	26·6	−10	10	approx. 40	…	5	5
PF–2	15	28·1	29·6	−20	15	approx. 60	20	25	5
	20	29·7	29·2	−20	5	75	20	30	30
PF–3	15	29·4	29·6	−20	15	45	10	15	40
	20	31·7	32·4	−40	40	40	0	30	40
PF–4	15	27·7	…	−20	25	40	…	…	40
	20	27·7	…	−35	10	−10	…	…	…
R1	15	27·0	…	5	75	>140	…	…	…
FK	15	28·3	26·7	−20	15	40	15	…	…



(*a*) Showing grain structure and slag inclusions × 100 (*b*) Showing veining and precipitates within the ferrite grains × 900

Fig. 11—Microstructures of pearlite-free basic Bessemer steel from heat *PF*–1 ; 15-mm. plate, from centre of ingot as hot-rolled, sectioned parallel to rolling direction. Etched in (*a*) 3% HNO_3, (*b*) 1% HNO_3, in alcohol

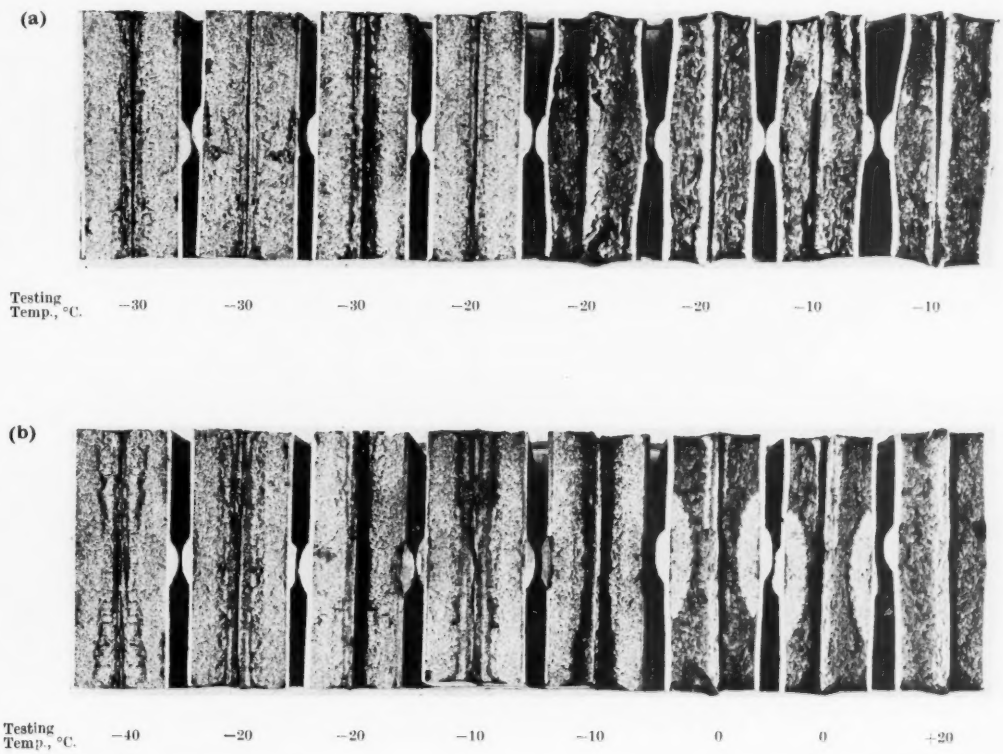


Fig. 12—Fractures of Kinzel tests on (*a*) pearlite-free basic Bessemer steel (heat *PF*–3), (*b*) fully killed carbon steel (heat *FK*)

higher for the pearlite-free steel than for the other two.

Figure 11 shows the microstructure at the centre of a 15-mm. plate taken from heat *PF*–1 and sectioned parallel to the rolling direction.

DUCTILITY AS WELDED

In several cases it has been found that brittle fracture in service has been caused by local formation of martensite, resulting from rapid cooling of the base material adjacent to the weld bead. This occurs most easily in the case of thin beads deposited on heavy plates. Efforts have been made to eliminate the risk of such martensite formation by limiting the manganese and carbon contents in the steel to certain values, which in turn are dependent upon the actual welding conditions.[6] This embrittlement hardening may also occur accidentally when hand-welding steels that have fairly low carbon and manganese contents and are normally quite weldable, but with pearlite-free steel no such risk arises. To illustrate this the following experiment was carried out.

Test pieces 12 mm. × 12 mm. × 55 mm. were cut from steels *PF*–4 and *FK* (*see* Table VII) and from a semi-killed steel containing 0·12% C, 0·01% Si, 0·41% Mn, 0·012% P, 0·025% S, and 0·007% N. These test pieces were heated to 1000° C. and quenched in a caustic soda solution, and Charpy bars prepared from them were tested in impact at different temperatures; the as-quenched hardnesses were also measured. Results are shown in Fig. 13. The two carbon steels increased considerably in hardness, compared with the pearlite-free steel. At the same time the impact values for the carbon steels were seriously lowered, whereas the pearlite-free steel shows the same energy absorption as in the original as-rolled condition (*cf.* Fig. 9*d*). The transition temperature is, in fact, somewhat lowered.

In addition to these risks of localized hardening, welding is generally accompanied by an embrittling effect, which is sometimes fatal to the construction; the nature of this effect is not fully understood at

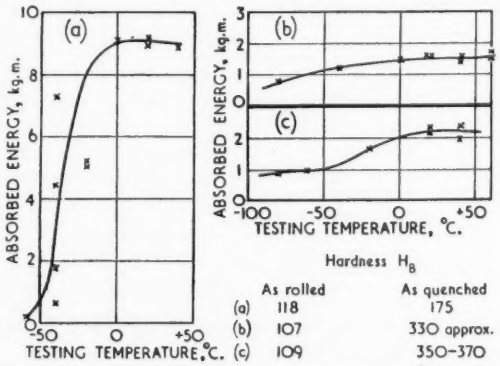


Fig. 13—Impact transition curves and hardness values for steels after quenching in caustic soda solution from 1000° C. (*a*) Pearlite-free basic Bessemer steel; (*b*) semi-killed electric-arc furnace steel; (*c*) fully killed electric-arc furnace steel

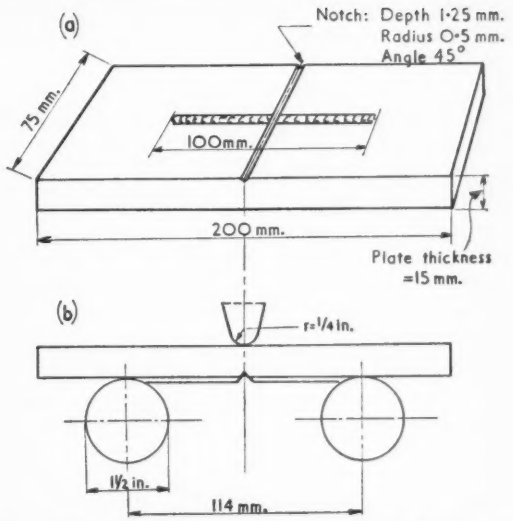


Fig. 14—Kinzel test.[10] (*a*) Dimensions of specimen; (*b*) bending procedure

present, but it is presumed to be inherent in certain regions of the heat-affected zone.[7, 8] To classify steels in respect of their tendency to this type of embrittlement, bend tests may be carried out on plates on which beads of weld metal are deposited under certain fixed conditions. In one method[9] no notch is used, whereas the Kinzel test[10] specifies a **V**-notch to be machined normal to the bead and to the bending direction. If these bend-tests are carried out at successively lower temperatures, a transition from ductile to brittle behaviour occurs in the same way as in the ordinary Charpy test; this implies that the transition temperature may be determined. Generally, if the welding has caused an appreciable embrittlement in the heat-affected zone, the resulting transition temperature will be comparatively high. However, this temperature depends very much on the ductility of the unwelded steel, and if no considerable embrittlement is developed by the welding the Kinzel test will probably classify steels in the same order as, for instance, the ordinary Charpy transition curves.

In this experiment the Kinzel test has been applied. The dimensions and testing procedure are shown in Fig. 14. Welding data were as follows:

Electrode	
Type	KB 5225 (Basic)
Diameter	5 mm.
Welding	
Speed	150 mm./min.
Voltage	26 V. (D.C.)
Current	175 amp.

The specimens were cooled or heated to the testing temperature before bending; temperature measurements showed that deviations due to heat exchange with the surroundings during the test were small. Figures 12*a* and *b* show the fracture surfaces obtained on testing pearlite-free steel *PF*–3 and fully killed carbon steel *FK* (*see* Table VII). The transition based on 50% shear fracture takes place, evidently, at roughly — 20° C. in the pearlite-free steel and between

— 10° and 0° C. in killed carbon steel. Even this comparison, therefore, is in favour of the pearlite-free steel.

CORROSION

No attempts have been made to measure the resistance of pearlite-free steel to atmospheric corrosion. It is expected, however, that it will behave very similarly to rimmed carbon steel, as the surface of such steel, also, consists of low-carbon ferrite. The higher phosphorus content in the pearlite-free basic Bessemer steel probably does not impede corrosion resistance. On the contrary, phosphorus is sometimes claimed to be beneficial in this respect.

Some preliminary stress-corrosion tests in nitrate solutions have shown that in this respect the pearlite-free steels are only slightly better than rimmed basic Bessemer steels. Like these and other low-carbon steels, therefore, they are not suitable for constructions in which there is a risk of this type of corrosion (*e.g.*, in boilers, etc.). Parkins' results,[11] according to which a very pronounced increase in stress-corrosion resistance occurs when the carbon content of the steel falls below 0·02%, have not been confirmed in this case.

CONCLUSIONS AND SUMMARY

From the foregoing results it appears that pearlite-free basic Bessemer steels with $< 0{\cdot}015\%$ C, about 0·1% P, and about 0·015% N are suitable for use as plates, structurals, beams, etc., for all general purposes where construction material with a yield strength of about 28 kg./sq. mm. is sufficient, provided that no special stress corrosion risks are involved. With regard to quality and properties, the following conclusions may be drawn:

(1) Pearlite-free steel has considerably lower impact transition temperatures than ordinary basic Bessemer steels, and in this respect, even after strain-ageing, it is equivalent to semi-killed or fully killed steels with about 0·15% C, 0·50–0·80% Mn, and low phosphorus and nitrogen contents.

(2) In welding, pearlite-free steel has certain advantages over plain carbon steels.

(3) The refining procedure is such that the oxygen content is continually limited to rather low values. The high phosphorus contents specified eliminate, in practice, the risks of overblowing that usually arise in normal basic Bessemer practice as soon as low phosphorus contents are pursued.

(4) Differences in composition and mechanical properties between various parts of an ingot are insignificant, because of the low tendency to segregation. The structure consists almost entirely of ferrite in equi-axed grains, and no substantial inferiority in directions normal to the rolling direction is to be expected.

Economically, the pearlite-free basic Bessemer steel is similar to ordinary basic Bessemer steel, but it offers the following advantages over the latter:

(5) The blowing is interrupted at a higher phosphorus content, which means a shorter blowing time and much smaller losses of iron and manganese in slag and exit blast, implying higher yield; moreover, a higher content of phosphorus in the slag is obtained.

(6) The optimum content of manganese is so low that in most cases the cost of ferro-manganese or spiegel additions is eliminated, at least when the manganese content of the pig iron exceeds 0·5%.

(7) The semi-killed character of solidification implies a high yield and low tapping costs.

Against these, however, there are the following drawbacks:

(8) The demand for low carbon content necessitates some precautions to be taken in steelmaking; in particular, a very low carbon content is required in all refractory material that comes into contact with the liquid steel. This, however, is a question of supervision rather than of expense.

(9) When the sulphur content is high, some difficulty may arise in hot rolling, owing to the low manganese content; this may call for a desulphurizing treatment of the pig iron.

Finally, it should be emphasized that ' semi-killed ' pearlite-free steels do not, of course, possess as good properties in respect of brittle fracture as do the fine-grained, aluminium-treated steels with, for instance, 0·12% C and 1% Mn. On the other hand, pearlite-free basic Bessemer steels may easily be aluminium-treated, in which case very low transition temperatures are obtained—sometimes appreciably lower than in a fine-grained carbon steel.

Acknowledgments

The author wishes to express his indebtedness to the Management of Stora Kopparbergs Bergslags AB, for permission to publish this paper, and to Professor Bo Kalling, Head of the Research Department, for continued support and helpful suggestions. Thanks are also due to Messrs. Börje Backström, Gösta Cedervall, Mats Falck, and Lennart Lindskog, at present or formerly members of the staff of the Research Department, who have conducted the experimental work, and further to Mr. Gunnar Ek of the Domnarfvet Steel Works, for the chemical analysis, especially the troublesome determinations of the low carbon contents.

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Deep-Drawing Properties of Sheet Steel

FUNDAMENTAL PRINCIPLES AND TEST METHODS

By Olov Svahn, Dr. Tech.

SYNOPSIS

A survey is given of a research project on deep-drawing of sheet steel, started in 1945 by the research organization of Jernkontoret.

The correspondence between practical deep-drawing figures from cupping operations is statistically analysed. The research work covered ten steel qualities.

The basic function of the deep-drawing process was studied using five materials very different in quality. The influence upon the drawing properties, characterized by the deep-drawing ratio, of various dimensions and other properties in cup drawing, was studied.

The Research Committee intends to propose a standard test for drawing quality of sheet steel. 932

IN 1945 a research project was started by Jernkontoret, designed

(i) To study existing methods of testing the deep-drawing properties of steel
(ii) To develop more reliable testing methods, if possible
(iii) To study the correspondence between test results and practice.

The Research Committee* has considered it appropriate to give a short review of the results of this work to date and of some especially interesting aspects of research in this field.

For many years manufacturers using the deep-drawing process have been anxious to find a method for testing sheet so as to be able to grade it for different purposes, to design their tools according to the properties of the sheet, and to discuss its deep-drawing properties with the supplier on the basis of reliable data. When the Committee began work, there was no generally accepted opinion about the value of the methods in use; it was only known that many manufacturers used methods based on extensive experience as a control for their sheets and to determine their suitability for various pressing or deep-drawing purposes. It was not known whether the test values could be reproduced, and there were no reliable data on the correspondence between test values or the possibility of using a certain material in a certain operation.

The Committee therefore began by studying the available literature. A summary was made of papers dealing with the various test methods, using the following classification:

(i) General aspects of deep-drawing and pressing
(ii) Tensile tests
(iii) Wedge-drawing tests
(iv) Tear-length tests
(v) Expanding tests (Siebel and Pomp)
(vi) Depression tests (*e.g.*, the Erichsen test)
(vii) Cupping tests (actual deep-drawing tests).

The present paper will not include a detailed description of the different testing methods published.[1] It may be observed, however, that the various investigators have tried in various ways to arrive at a test which corresponds as far as possible with the function of deep-drawing, but which is at the same time simple and preferably uses well-known material-testing methods. There are also few published results showing the relation between test figures and practical experience.

THE TWO PRINCIPLES OF DEEP-DRAWING

A theoretical study of the principles of plastic deformation in deep-drawing shows the reason for classifying the test methods as described in the previous section. The Committee first found that it was necessary to divide the deep-drawing methods used in practice into:

(i) Deep-drawing with pure elongation (Fig. 1*a*)
(ii) Deep-drawing with the material movement approximately following the punch in its linear movement (Fig. 1*b*).

In the former method deformation occurs under biaxial elongation (tangential and radial elongation

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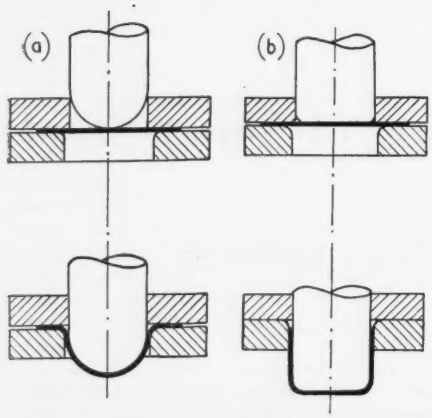


Fig. 1—Diagrammatic representation of deep-drawing (*a*) with pure elongation, (*b*) with the material movement approximately following the punch in its linear movement

under tangential and radial stresses). Theoretically, the outer parts of the blank can be fixed between the die and the blank holder so that no material sliding is possible from the fixed part to the parts under deformation. In these circumstances the reduction in thickness of the sheet corresponds theoretically to the elongation, with the exception of variations arising from the irregular form of the punch and the friction between punch and blank.

The second method is a much more complicated form of plastic deformation. Tangential and radial elongation (*i.e.*, biaxial elongation) take place in the bottom of a cylindrical drawn piece. There is tangential strain over the punch radius. Where the cylindrical part begins there is compression instead of elongation. The radial elongation in the bottom increases from the centre to the periphery. Along the generatrix up to the top of the cup there is an increase in radial elongation and tangential compression. Normally, when the clearance between punch and die is large enough, these are the only types of strain. The compression increases more than the elongation, and so the thickness of the material increases towards the top by 30–40% or more. The parts of the blank still sliding between die and blank holder are subject to pressure perpendicular to the blank surface. This pressure must be sufficient to eliminate wrinkles on the blank. The force that the punch has to work against results from the friction forces between the sheet and the die and between the sheet and the blank holder, plus the resistance from the material resulting from compression under the blank holder, bending over the die radius, and straightening when the material leaves the die radius. This means that there must be a radial elongation effect on the blank. This can be observed and recorded with a device which simultaneously registers the linear movement of the punch and the linear movement of a certain point on the periphery of the blank. It can be shown that, under the same tool and drawing conditions, different materials give different values for this elongation effect (Fig. 2).

Thus, the complex nature of the deformation caused by deep-drawing with the material sliding can hardly be explained by figures derived from a simple test method. The Committee has also found that the test method in this case must be a functional test (a technological test), where the conditions are as near manufacturing conditions as possible. For deep-drawing with pure elongation, however, any kind of

Table I
MATERIAL DATA

Material	Chemical Composition, %								Mechanical Properties				Average Grain Size, μ
	C	Si	Mn	P	S	Al (sol.)	Al (insol.)	N	Yield Point, tons/sq. in. (kg./sq. mm.)	Tensile Strength, tons/sq. in. (kg./sq. mm.)	Elongation, %		
											10 × dia. δ_{10}	25 × dia. δ_{25}	
A	0·08	0·01	0·33	0·004	0·021	0·041	0·007	0·016	8·6– 9·4 (13·5–14·7)	17·4–18·2 (27·3–28·5)	50·7–58·4	39·0–44·5	25–40
B	0·06	Trace	0·31	0·005	0·025	...	...	...	13·5–16·8 (21·3–26·4)	18·8–20·3 (29·5–31·9)	41·4–43·4	32·8–34·2	25–40
C	0·07	Trace	0·25	0·006	0·019	...	...	0·007	13·7–15·2 (21·5–23·8)	19·2–19·5 (30·1–30·5)	44·7–51·5	35·2–39·9	25–30
D	0·06	Trace	0·32	0·053	0·039	...	...	...	16·7–18·1 (26·3–28·3)	24·2–25·0 (37·8–39·3)	26·3–32·2	18·7–23·7	40–60
E*	...	...	...	...	...	...	...	...	...	...	...	...	...
F	0·06	Trace	0·16	0·017	0·011	...	...	...	...	23·0–24·5 (36·1–38·4)	20·4–29·5	12·0–16·9	20–25
G	0·12	Trace	0·19	0·018	0·019	...	...	...	...	25·3–26·1 (39·6–41·9)	15·6–26·7	7·7–14·0	15–25
H	0·07	0·04	0·18	0·004	0·009	...	...	...	17·6–18·5 (27·5–29·0)	20·2–21·1 (31·6–33·2)	34·2–44·6	24·1–32·1	30–40
I	0·12	0·10	0·42	0·013	0·012	...	...	...	23·9–26·2 (37·5–41·2)	30·5–30·8 (47·8–48·4)	22·0–22·9	14·2–15·2	15–20
J	0·14	0·10	0·36	0·018	0·023	...	...	...	20·6–21·5 (32·3–33·3)	24·9–26·0 (39·1–40·8)	23·4–29·1	13·9–17·7	25–40

* Owing to an accident, no reliable figures for material *E* are available

biaxial elongation method can be used as a functional test.

RESEARCH CARRIED OUT BY THE SWEDISH COMMITTEE

The Committee has been working along the following lines. Deep-drawing tests have been carried out using the two methods described in the previous section, and the basic problem of the conditions under which the test results can be reproduced has been studied. The dispersion indicates the number of tests necessary to give a deep-drawing figure. These figures have then been compared with the results from the most acceptable published tests. An attempt has also been made to find some simple function in the actual deep-drawing tests that would give an acceptable classification of different materials.

When a method was found that was suitable for proposing as a standard deep-drawing test, it was studied intensively using a number of different deep-drawing materials.

Materials Used

It was first necessary to obtain a number of sheet steels with widely differing deep-drawing characteristics from the practical standpoint. Three Swedish manufacturers of deep-drawing steel each supplied three batches, grading them ' very good,' ' normal,' and 'bad.' For comparison, an American steel, known to be of good deep-drawing quality, completed the group. The first tests were carried out under normal working conditions in a press shop. The chemical and physical properties of the materials were analysed in the suppliers' laboratories. At the same time, some preliminary deep-drawing tests were carried out as cup tests at the Royal Institute of Technology in Stockholm. The characteristics of the materials as described by the laboratories are given in Table I.

Test Results

Both in the shop tests and in the cup tests at the Institute of Technology a deep-drawing criterion (the ' deep-drawing ratio ' D/d) was used. This is the relation between the diameter of the blank and the diameter of the punch, or tool diameter.

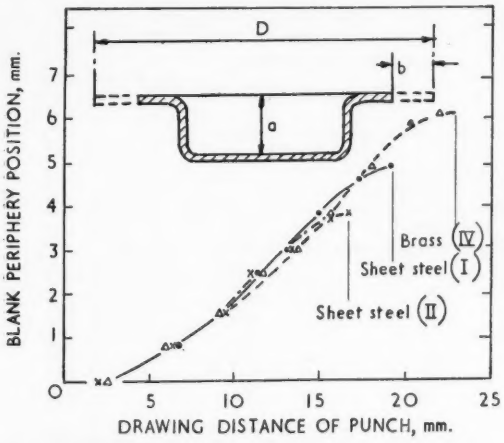


Fig. 2—Elongation effect for different materials

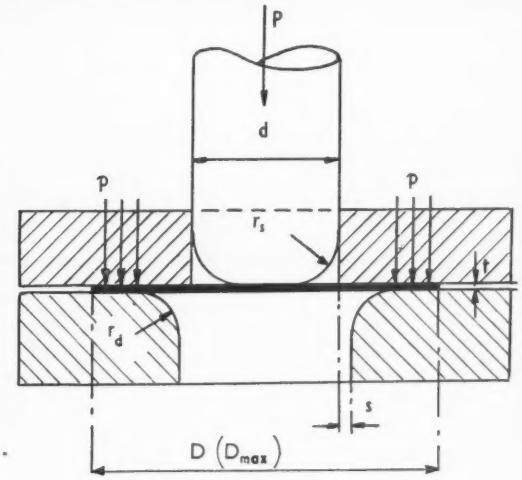


Fig. 3—Cup test

To explain the drawing conditions the following symbols are used (Fig. 3):

D = Blank dia.
D_{max} = Greatest dia. of blank which can be drawn without fracture
d = Punch dia. (tool dia.)
r_s = Edge radius of punch
r_d = Edge radius of die
s = Clearance (radial distance between punch and die hole)
P = Drawing force (pressure of punch)
p = Blank-holder pressure
H = Surface roughness of functioning surfaces on the tool
t = Thickness of drawn material.

Table II shows the difficulties of obtaining a classification of the group of materials tested. The different materials are marked A, B, C, etc.; the rank is 1, 2, 3, etc., from the best to the worst; and the different test series are marked I, II, III, etc. Table II also shows the rank correlation of the various test series.

The rank correlation coefficient is a value for the degree of the correspondence between the ranks of two test series. It is based upon a statistical calculation.[2] The significance is also statistically calculated in relation to the number of components in the series and the statistical accuracy desired.

The rank correlation coefficients are very bad: only three out of 31 comparisons give a coefficient $\geqslant 0{\cdot}75$. Most values are very small and even negative. Only the three values over $0{\cdot}75$ show a significant correlation from a statistical standpoint. From a practical standpoint even these values are not great enough. The actual differences between the characteristics of the steels should be large enough to give a complete significance in rank correlation, *i.e.*, a coefficient of 1, between tests I, II, III, IV, and V, respectively, and between tests VI and VII. The coefficient value of $0{\cdot}92$ between tests VI and VII is of especial interest. A good correspondence may be expected between deep-drawing with pure elongation and the Erichsen tests. Remembering that variations in values from the Erichsen tests are due to a lack of exact definitions of the method, it is evident that a near relationship exists between the two methods mentioned. This

Table II

RANKS IN DRAWING TESTS AND RANK CORRELATION COEFFICIENTS

Rank	Cup Drawing					Stretch Drawing	
	I	II	III	IV	V	VI	VII
	Factory Test: Tool Dia. 140 mm.	K.T.H.*: Tool Dia. 58 mm.	K.T.H.: Tool Dia. 37 mm.	K.T.H.: Tool Dia. 25 mm.		Factory Test: Tool Dia. 140 mm.	Erichsen Test
				June, 1946	Oct., 1946		
1	A	E	A2	A	A2	B	A
2	C	A	J	B	H	C	C
3	F	F	H	H	F	A	B
4	I	H	I	C	E	D	D
5	B	I	E	E	B	E	F
6	E	J	F	D	C	F	H
7	G	G	G	F	J	H	E
8	H	B	C	I	I	G	G
9	J	C	B	J	D	I	I
10	D	D	D	G	G	J	J

Rank Correlation Coefficients†

$r12 = 0.33$	$r15 = 0.48$	$r23 = 0.72$	$r26 = -0.22$	$r35 = 0.50$	$r45 = 0.73$	$r56 = 0.33$
$r13 = 0.18$	$r16 = 0.43$	$r24 = 0.16$	$r27 = -0.14$	$r36 = -0.49$	$r46 = 0.78$	$r57 = 0.47$
$r14 = 0.42$	$r17 = 0.54$	$r25 = 0.68$	$r34 = 0.05$	$r37 = -0.28$	$r47 = 0.81$	$r67 = 0.92$

* K.T.H. = Royal Institute of Technology
† r12, etc. = Rank correlation coefficient between grades 1 and 2, etc.

has also been one of the factors influencing the Committee in framing a test for materials used in deep-drawing with pure elongation.

To enable the correlation between the results mentioned above and values representing other characteristics of the materials to be studied, a large number of accurate tests have been carried out. The following characteristics have been studied:

(i) Elongation, yield point, and tensile strength in ordinary tensile tests
(ii) Strain at maximum load in tensile tests
(iii) Vickers hardness
(iv) Increase in wall thickness
(v) Increase in hardness
(vi) Erichsen test values
(vii) Wedge-drawing values (wedge-drawing Erichsen test)
(viii) Cupping-test values (Erichsen test).

Moreover, tests have been carried out to indicate how the rank can be reproduced, what variations occur when another tool diameter is used, and the values obtained from two-stage drawing.

The results are given in Tables III–XI. Statistically calculated rank correlation figures are given for each test using the rank order derived from the values obtained with the 25-mm. die, carried out in the first test series at the Institute of Technology, Stockholm. The testing conditions are noted in each Table.

Furthermore, an attempt has been made to find the correlation of the depth of the cups with the point at which wrinkles start on the walls. It has been suggested that such values are useful for determining how suitable a material is for deep-drawing purposes. The results are given in Table XII.

DISCUSSION OF DIFFERENT TESTING METHODS

A theoretical discussion of certain ' deep-drawing tests ' (*e.g.*, those in classes iv, v, and vi on p. 129) support the opinion that they are unsuitable for classifying the materials for practical purposes. None of them takes into consideration the characteristics of deep-drawing conditions, and they are sometimes impossible to carry out with the desired accuracy.

These test methods in no way represent the same type of stresses in the material as in the drawing operation, especially those based on a rupture function starting from a hole or a slit. In addition, the influence of punching or machining on the material (strain hardening and influence on the structure) cannot be avoided and is not taken into consideration.

The Committee carried out a test series on these questions, but the results showed that no acceptable

Table III

VALUES FOR CALCULATING RANK CORRELATION COEFFICIENTS

Deep-drawing tests made at K.T.H., June, 1946

Machine: Hydraulic press
Tool: Die dia., mm. $d_1 = 25 + 0.02 - 0$
Punch dia., mm. $d_1 = 22.78$ (*A*) and 22·40 (others)
Edge radius, mm.: die $r_d = 10 \pm 0.1$
" " " : punch $r_s = 8 \pm 0.1$
Blank-holder pressure (spring pressure), kg./sq. cm. $p = 10 \pm 1$
Lubrication: Motor oil
Drawing Speed, mm./sec. About 55
Number of specimens per blank diameter 3
Stage between diameters, mm. 0·25

Rank	Material	D_{max}/d
1	A	2·450
2	B	2·305
3	H	2·300
4	C	2·280
5	E	2·275
6	D	2·255
7	F	2·235
8·5	I	2·225
	J	
10	G	2·190

Table IV

'CONVENTIONAL' STRESS/STRAIN VALUES

Standard specimen width

Rank	Upper Yield Point $\sigma_{s\ddot{o}}$			Tensile Stress σ_B			Elongation $10 \times$ dia. δ_{10}		Elongation $25 \times$ dia. δ_{25}	
	Material	tons/sq. in.	kg./sq. mm.	Material	tons/sq. in.	kg./sq. mm.	Material	%	Material	%
1	A	9·0	14·1	A	17·8	27·9	A	54·4	A	41·8
2	C	14·7	23·0	C	19·3	30·3	C	48·0	C	37·1
3	B	15·5	24·4	B	19·9	31·1	B	42·4	B	33·4
4	D	17·3	27·1	H	20·7	32·5	H	39·9	H	27·4
5	H	18·1	28·4	F	23·5	36·9	D	29·5	D	22·0
6	J	20·8	32·7	D	24·4	38·3	J	27·1	J	15·9
7	I	25·3	39·6	J	25·6	40·1	F	25·9	I	14·8
8	...	...	...	G	26·0	40·8	I	22·5	F	14·6
9	...	...	...	I	30·6	48·1	G	20·4	G	10·3

Rank Correlation Coefficients
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm.: $\sigma_{s\ddot{o}}$ $r = 0{\cdot}812$
" " " σ_B $r = 0{\cdot}904$
" " " δ_{10} $r = 0{\cdot}921$
" " " δ_{25} $r = 0{\cdot}896$

Table V

ELONGATION AT MAXIMUM LOAD

Width of specimen: Standard
Length of specimen: 270 mm. (***A***, ***B***, ***C***, and ***D***), 150 mm. (***F*** and ***G***), 100 mm. (***H***, ***I***, and ***J***)

Rank	Elongation at Max. Load		$\frac{\delta_{\text{max. load}}}{\delta_{10}} \times 100$	
	Material	%	Material	%
1	A	23	D	49
2	B	17	A	42
3	C	16·7	B	40
4	D	14·3	C	38
5	I	6·7	I	30
6	F	5·7	F	22
7	H		J	19
8	J	5	G	18
9	G	3·7	H	14

Rank Correlation Coefficients
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm.: Elong. at max. load $r = 0{\cdot}825$
" " " $\frac{\delta_{\text{max. load}}}{\delta_{10}} \times 100$ $r = 0{\cdot}487$

Table VI

VICKERS HARDNESS OF TEST MATERIAL

Rank	Material	Hardness, D.P.N.
1	A	89·5
2	B	99·5
3	C	108·5
4	H	117·0
5	D	123·0
6	F	124·5
7	E	125·5
8	G	132·0
9	J	138·0
10	I	159·0

Rank Correlation Coefficient
Deep-drawing tests, K.T.H., June 1946
Tool dia. 25 mm.: Hardness $r = 0{\cdot}912$

Table VII

HARDNESS INCREASE IN CUPS

Specimens drawn at $D_{\max}/d$ (deep-drawing, K.T.H., June, 1946)

Rank	Material	Hardness Increase, %
1	D	31·8
2	B	25·7
3	A	25·2
4	E	20·4
5	C	18·1
6	H	15·1
7	I	13·6
8	J	9·8
9	G	9·0
10	F	6·5

Rank Correlation Coefficient
Deep-drawing tests, K.T.H., June 1946
Tool dia. 25 mm.: Hardness increase in cups $r = 0{\cdot}683$

Table VIII

WALL-THICKNESS VARIATION IN CUPS

Tool: Specimens drawn at $D_{\max}/d$ (deep-drawing, K.T.H., June, 1946)

Rank	Thickness Decrease		Thickness Increase	
	Material	%	Material	%
1	A	16	D	16
2	H	15	A	
3	I	14	B	18
4	B		C	20
5	C	13	I	24
6	E	12	H	27
7	F	10	F	29
8	G	8	G	32
9	J	7	E	
10	D	6	J	34

Rank Correlation Coefficients
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm.: Thickness decrease $r = 0{\cdot}694$
" " " Thickness increase $r = 0{\cdot}639$

Table IX
ERICHSEN CUP TEST

Machine : Original Erichsen cup-test apparatus

Tool :
Punch dia., mm. = 33
Die dia., mm. = 35·6 (***D***), 35·2 (***A***), and 35·4 (others)

Blank-holder pressure, kg.: 800 (blank dia. 66 mm.) and 900 (blank dia. 70 mm.)

Blank dia., mm. 66 and 70

Lubrication: Tallow

Classification: Figured by rupture

Rank	Fagersta		Munkfors	
	Material	Average D_{max}/d (3 Specimens)	Material	Average D_{max}/d (3 Specimens)
1	H	>2·12	J	>2·00
2	E		F	
3	A		G	
4	C	2·12	H	1·91
5	J		A	1·89
6	B	2·10	E	1·81
7	D	2·08	B	1·76
8	F	2·04	I	
9	G	2·03	C	1·75
10	I	2·02	D	1·73

Rank Correlation Coefficients
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm.: D_{max}/d (Fagersta) $r = 0·606$
" " " D_{max}/d (Munkfors) $r = 0·342$

correlation exists between the tests (classes iv, v, and vi on p. 129) and pure deep-drawing.

The correlation values given show that there is no correlation good enough to make it possible to use any of these tests in practice for grading the different materials according to their suitability for deep-drawing. The tests must be made as similar as possible

Table X
ERICHSEN WEDGE-DRAWING TESTS

Specimen dimensions according to Fig. 4
Reduction of width controlled with marks on the specimen
Bulge tests with the Erichsen equipment with punch dia. of 14 mm.
Results compared with Erichsen tests in original condition of the sheet

Rank	Sandviken		Domnarfvet	
	Material	Angle Coefficient	Material	Angle Coefficient
1	D	1·50	D	0·50
2	I	0·46	I	
3	B	0·44	B	0·48
4	A	0·38	C	0·29
5	J	0·37	A	
6	F	0·36	J	0·27
7	C	0·34	E	0·17
8	E	0·32	H	0·15
9	G	0·25	F	0·09
10	H	0·19	G	0·08

Rank Correlation Coefficients
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm.: Angle coefficient (Sandviken) $r = 0·04$
" " " Angle coefficient (Domnarfvet) $r = 0·38$

Table XI
DEEP-DRAWING IN TWO STAGES

Machine: Hydraulic press
Tools for 1st stage:
Punch dia., mm. = 35 (for ***A*** = 35·4)
Die dia., mm. = 37·6
Die edge radius, mm. = 10
Number of blanks per stage = 3
Stage interval, mm. = 0·5
Tools for 2nd stage:
Punch dia., mm. = 25·3
Die dia., mm. = 30·1
Edge broken to 45°, radius 1·5 mm.
Lubrication Normal motor oil

Rank	Material	D/d_1
1	A	2·80
2	I	
3	J	2·78
4	G	
5	F	
6	H	2·76
7	E	2·74
8	C	2·70
9	B	2·69
10	D	2·65

Rank Correlation Coefficient
Deep-drawing tests, K.T.H., June, 1946
Tool dia. 25 mm. : Deep drawing in two stages $r = 0·23$

to shop practice (*i.e.*, cup deep-drawing or ' cupping '). The values also show that even the real deep-drawing test is influenced by unknown but obvious variations in testing conditions, which means that reproduction is difficult.

This fact is probably one of the most important, as it is of general application. This can be seen especially in the Erichsen tests. A statistical calculation based on values from Erichsen tests made on the same materials at a number of different laboratories, all of which were very familiar with this type of testing, gave a reproducibility with a probability of less than 1%. All the tests were made under the testing conditions normal to each laboratory. The directions for using the apparatus allow such a wide dispersion. A further description of these tests will be made and published in the future. The Committee's

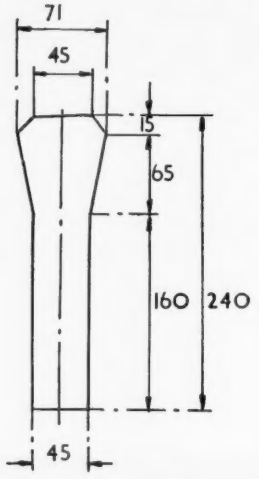


Fig. 4—Test piece for wedge-drawing test

Table XII

DEEP-DRAWING WITHOUT BLANK HOLDER

***Machine*:** Hydraulic press
***Tool*:** Die dia., mm. = 37·6
Punch dia., mm. = 35·0
Edge radius (die), mm. = 10
Edge radius (punch), mm. = 8
***Lubrication*:** Motor oil
***Drawing speed*, mm./sec.:** About 5
***No. of specimens per blank dia.*:** 3
***Stage between diameters*, mm.** 0·5

Rank	Material	D_{max} for Cup without Wrinkles, mm.
1	D	61·00
2	C	60·00
3·5	B, E	59·50
5·5	G, A	59·25
8·5	H, J, I, F	59·00

Rank Correlation Coefficient
Deep-drawing tests, K.T.H., June 1946
Tool dia. 25 mm. : D_{max} for cup without wrinkles
$r = 0.355$

opinion is that the Erichsen test is so well known that this criticism of it will be of great interest in future discussions.

One of the main points in the Committee's study of the conditions of the deep-drawing function is the lack of reproducibility, even in test series made under very uniform conditions. In the actual deep-drawing tests, for instance, only one factor, the diameter, is altered, or drawing is done in two stages instead of one. The interest thus concentrates on the results from tests with tools related in scale and on the influence of variations in different factors, *e.g.*, die- and punch-edge radius, clearance, diameter, and surface finish, as well as drawing speed, blank-holder pressure, lubrication, wear on the tools, etc. Unfortunately research was not based on a statistical examination of the problem from the start.

EXPERIMENTS WITH WIDELY DIFFERING TYPES OF MATERIAL

To study these points more closely, the Committee decided to deal next with five materials with very different characteristics from the deep-drawing point of view. This decision was based on a theoretical calculation. The fundamental characteristics which make sheet and strip suitable for deep-drawing seem to be structural and mechanical. In the microstructure grain size and form are presumed to be important. The distribution of structural phases and that of segregations also exert an influence.

Furthermore, the existence of preferred orientations has to be taken into consideration, as has been shown clearly by Jackson, Smith, and Lankford,[3] who have constructed a figure for the plastic anisotropy to indicate the relationship between the deep-drawability of the material in different directions.

Among the mechanical properties the true-stress/true-strain diagram, characterized by the function

$$\sigma = k \times \delta^n$$

where σ = True stress
δ = True strain
k = Constant
n = Strain-hardening exponent,

seems to be more fundamental for judging the deep-drawing properties than other common mechanical-test values.

Against the background of these statements it was found convenient to continue the basic research work with the following materials:

'Extra deep-drawing sheet steel' (of American origin)
A Swedish deep-drawing steel with different characteristics from the American sheet
18/8 stainless steel
Brass
Aluminium.

Table XIII gives the analysis and mechanical properties of these materials.

The tests were partly carried out at the Stockholm Institute of Technology and partly in the Machinability Research Department of Sandviken Steel Works. In the following description of results for

Table XIII

MATERIAL DATA

Material		Chemical Composition, %											Mechanical Properties				Average Grain Size, μ
													Yield Point, tons/sq. in. (kg./sq. mm.)	Tensile Strength, tons/sq. in. (kg./sq. mm.)	Elongation, %		
No.	Type	C	Si	Mn	P	S	Cr	Ni	Al	Cu	Pb	Fe			5 × dia. δ_5	10 × dia. δ_{10}	
I	Deep-drawing sheet steel (American)	0·052	Trace	0·32	0·002	0·011	0·02	0·03	0·002	...	...	...	15·2 (23·8)	20·7 (32·6)	59·2	46·1	50
I	Deep-drawing steel (Swedish)	0·10	0·18	0·36	0·013	0·010	0·07	0·06	...	...	...	...	20·2 (31·7)	27·0 (42·3)	45·8	36·6	35
III	18/8 stainless steel	0·12	0·39	0·41	0·016	0·011	18·5	8·7	...	...	...	...	19·8 (31·0)	47·5 (74·3)	73·3	63·8	35
IV	Brass	...	...	...	...	...	...	...	...	62·7	0·32	...	8·3 (13·0)	22·5 (35·3)	62	55	25
V	Aluminium	...	0·09	...	...	...	...	...	...	...	...	0·26	2·2 (3·4)	5·0 (7·8)	55	44	50

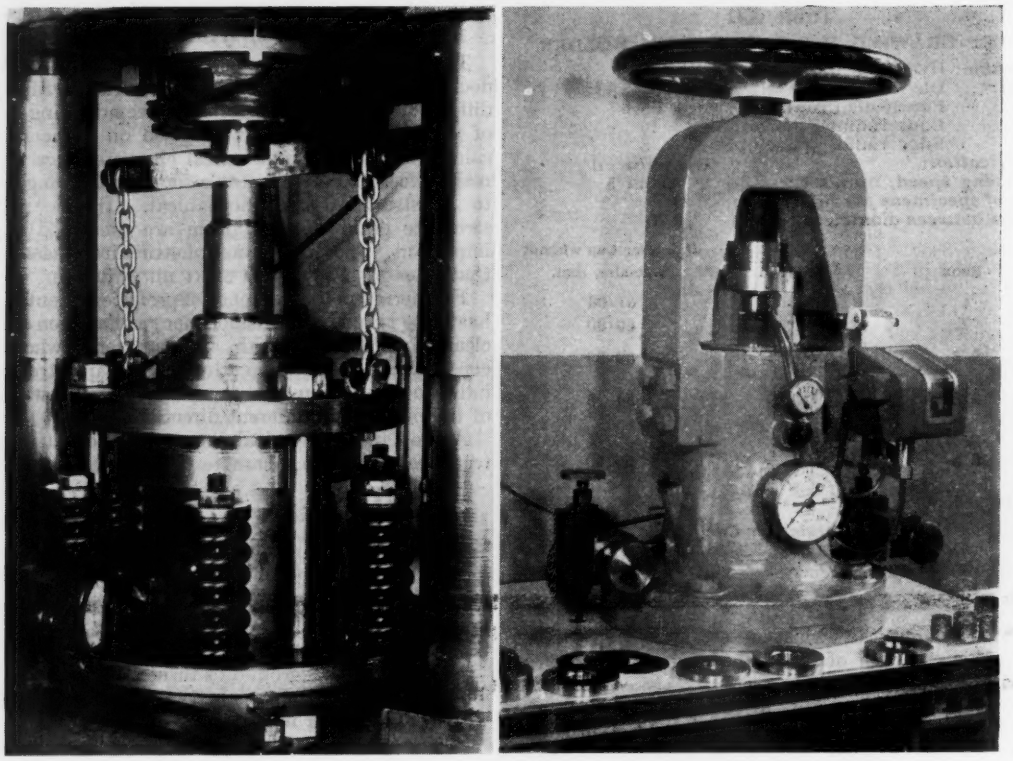


Fig. 5—Hydraulic presses used at (*a*) the Institute of Technology, (*b*) Sandviken

individual factors in the testing operation the chronological sequence of the tests, the sometimes confusing results, and the means of overcoming the difficulties are disregarded.

Testing Equipment

At the Institute of Technology a hydraulic press (Fig. 5*a*) was used; its details are as follows:

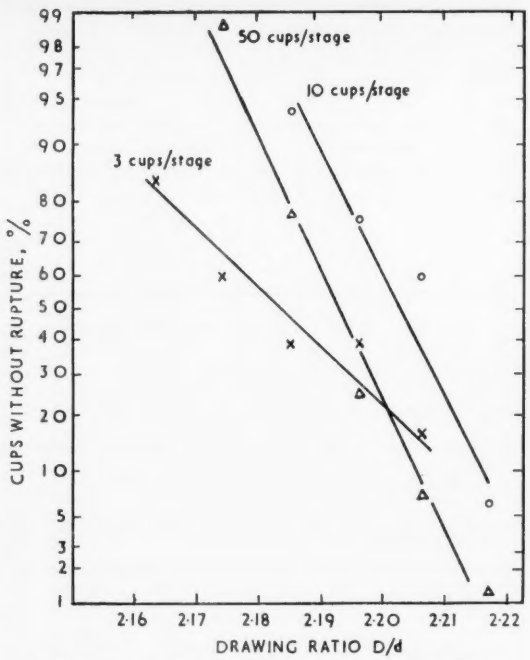
Maximum pressure: 100 tons
Speed range: 0–0·59 in./sec. (0–15 mm./sec.)
Possibilities of regulation: (i) Constant speed, varying pressure; (ii) Constant pressure, varying speed
Method of applying blank-holder pressure: Spring control
Maximum blank-holder pressure: 2·6 tons.

The tools, with varying diameters, radii r_s and r_d, clearances, and surface finish, are shown in Fig. 3. A device for drawing-force measurements is fitted to the punch holder.

Details of the hydraulic press at Sandviken (Fig. 5*b*) were as follows:

Maximum pressure: 12 tons
Speed range: 0–0·47 in./sec. (0–12 mm./sec.)
Possibilities of regulation: (i) Constant speed, varying pressure; (ii) Constant pressure, varying speed
Method of applying blank-holder pressure: Hydraulic control
Maximum blank-holder pressure: 7·3 tons.

The tools are the same as those at the Institute of Technology. Equipment for measuring drawing force is mounted in the punch holder with recording apparatus of special design.



d = 23 mm., r_s = 8 mm., r_d = 4·5 mm., s = 1·20 mm., t = 1·00 mm.

Fig. 6—Distribution diagram for sheet steel

Table XIV

PROPORTION OF CUPS WITH RUPTURE AT DIFFERENT NUMBERS OF BLANKS PER STAGE

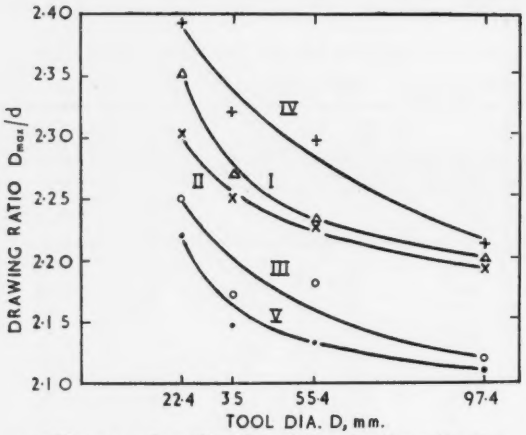
It is assumed that each percentage figure of cups without prior rupture has the same probability

No. of Cups without Rupture	No. of Cups with Rupture	Average Cups without Rupture, %	90% Probability that the Per Cent. Figure of Cups without Rupture is		No. of Cups without Rupture	No. of Cups with Rupture	Average Cups without Rupture, %	90% Probability that the Per Cent. Figure of Cups without Rupture is	
			Greater Than	Less Than				Greater Than	Less Than
		3 cups per stage			39	11	77	69	84
3	0	84	57	97·4	38	12	75	67	82
2	1	61	32	86	37	13	73	65	81
1	2	39	14	68	36	14	71	63	79
0	3	16	2·6	43	35	15	69	61	77
					34	16	67	59	75
		6 cups per stage			33	17	65	57	74
6	0	91	72	98·5	32	18	63	55	72
5	1	77	55	92	31	19	62	53	70
4	2	64	41	83	30	20	60	51	68
3	3	50	28	72	29	21	58	49	66
2	4	36	17	59	28	22	56	47	64
1	5	23	8	45	27	23	54	45	63
0	6	9	1·5	28	26	24	52	43	61
					25	25	50	41	59
		10 cups per stage			24	26	48	39	57
10	0	94	81	99	23	27	46	37	55
9	1	85	69	95	22	28	44	36	53
8	2	76	58	90	21	29	42	34	51
7	3	67	49	83	20	30	40	32	49
6	4	59	40	76	19	31	38	30	47
5	5	50	32	68	18	32	37	28	45
4	6	41	24	60	17	33	35	26	43
3	7	33	17	51	16	34	33	25	41
2	8	24	10	42	15	35	31	23	39
1	9	15	5	31	14	36	29	21	37
0	10	6	1	19	13	37	27	19	35
					12	38	25	18	33
		50 cups per stage			11	39	23	16	31
50	0	98·7	95·6	99·8	10	40	21	14	29
49	1	96·7	92·5	99·0	9	41	19	13	26
48	2	94·8	90	97·8	8	42	17	11	24
47	3	92·9	87	96·5	7	43	15	9·4	22
46	4	91·0	85	95·2	6	44	13	7·7	20
45	5	89	83	93·7	5	45	11	6·3	17
44	6	87	80	92·3	4	46	9·0	4·8	15
43	7	85	78	90·6	3	47	7·1	3·5	13
42	8	83	76	89	2	48	5·2	2·2	10
41	9	81	74	87	1	49	3·3	1·0	7·5
40	10	79	71	86	0	50	2·3	0·2	4·4

Results of the Tests

Cup-drawing tests were made with observations of the drawing ratio. At first observations were made at two values of the deep-drawing ratio: the point where the drawing ratio reaches the limit at which rupture starts, and the point where all cups ruptured. In subsequent tests it was decided that only the former was important for determining the suitability of the different materials for deep-drawing purposes. When the deep-drawing ratio is increased to the point at which rupture starts, the limit is reached where there is a danger of rejects in production. It was found that no better accuracy was obtained by striving for the upper point.

The dispersion in the testing values was studied statistically in series of 3, 6, 10, and 50 tests, respectively, under very uniform conditions in regard to all possible influencing factors, except that of variations in the material itself. The dispersion in test results is, under these conditions, the sum of systematic defects and influence of variations in mechanical conditions over which there is no means of control. There is a normal dispersion in the test series, characterized by the normal distribution diagrams (Fig. 6). The correspondence with a theoretical probability calculation is thus also good (*see* Table XIV). This means that normal dispersion is to be expected under exactly controlled test conditions, and the number of tests can be fixed for a certain probability of obtaining all cups without rupture. From all practical purposes (test series, costs, time saving, etc.) six tests will be sufficient. According to Table XIV the average percentage of cups without rupture can be fixed at about 90% when the point at which rupture starts is found. Each stage means a difference in blank diameter of about 0·5%. The accuracy is very little increased with more than six tests, and a great increase in the number of tests is required to obtain accuracy a few per cent. higher.



r_d = 10 mm., r_g = 8 mm. (d = 22·4 and 35 mm.) or 10 mm. (d = 55·4 and 97·4 mm.), s = 1·30 mm., t = 1·00 mm.

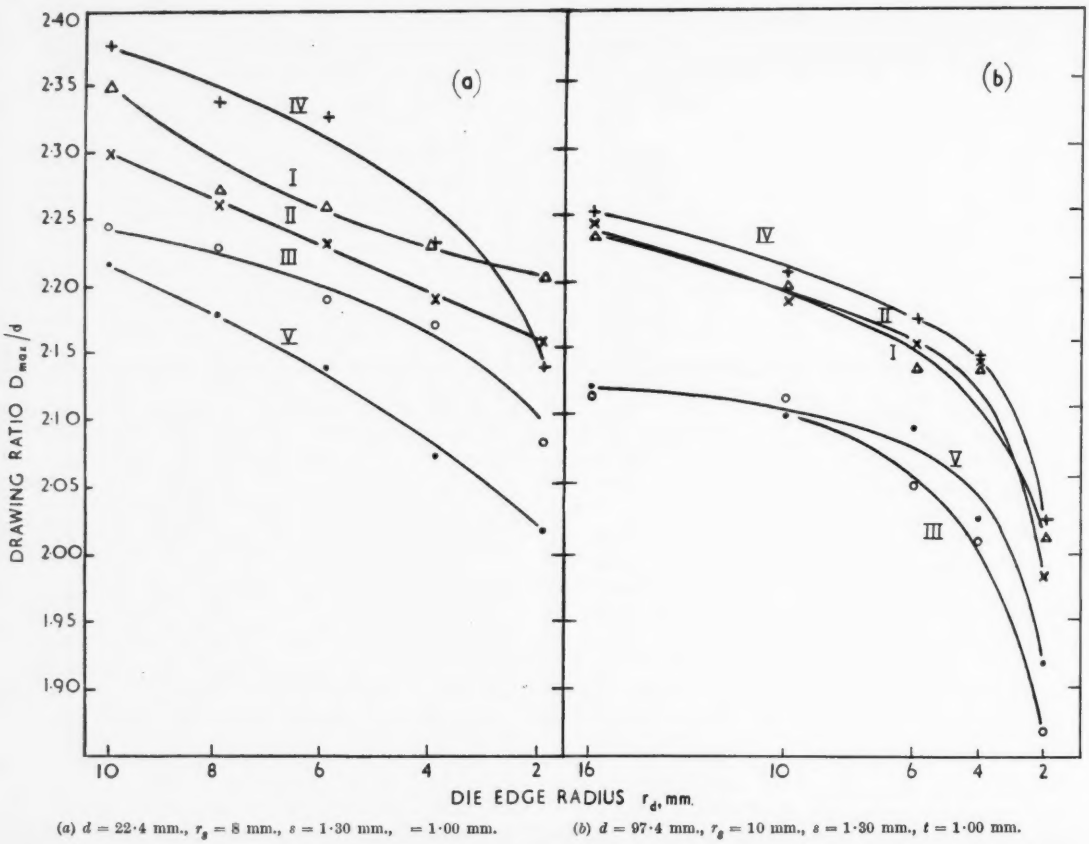
Fig. 7—Drawing ratio as a function of tool diameter

Variations in material properties also show up in the results, but these are, from experience with the distribution diagrams, also of normal distribution type. This aspect of the problem is left for future research work.

Tool Size—The size of the tool and its influence on the value of the deep-drawing ratio and classification of the different materials is being intensively studied. It has been shown that increasing tool diameter decreases the drawing ratio (Fig. 7). The rank is regular, except for the curve representing brass, where there are some irregularities in a number of test series. No explanation for this has yet been found. The steep inclination of the curves for smaller tool diameters is in agreement with the fact that the edge radius is fixed for all diameters, and is therefore abnormally great for smaller tools. Over a certain diameter/edge-radius ratio the curves become approximately straight lines with a characteristically slow rise.

Within the tool size ranges tested the sequence for various materials at a certain size is significant for all the other sizes, taking into account the practical need for accuracy.

Edge Radius—The edge radius is, according to many investigations,[4] of extreme importance for the result of a deep-drawing operation. Edge radius is, for the purposes of this discussion, the radius in both the punch and the die. The Committee's research work covers both equal variations in edge radius in punch and die, and a constant radius on the former with a varying radius on the latter. Roughly the influence on the deep-drawing ratio is exerted from



(*a*) d = 22·4 mm., r_g = 8 mm., s = 1·30 mm., = 1·00 mm. (*b*) d = 97·4 mm., r_g = 10 mm., s = 1·30 mm., t = 1·00 mm.

Fig. 8—Drawing ratio as a function of die edge radius

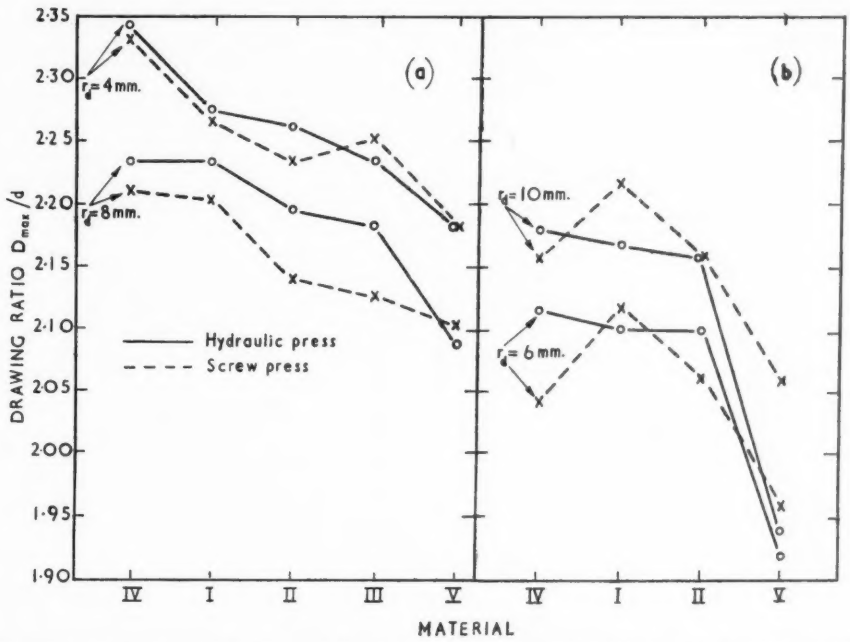
Fig. 9—Cups drawn to produce wrinkles: (*left*) wrinkles starting to form; (*right*) drawing completed

both parts of the tool. However, the limiting factor varies. When the radius reaches small values in the punch, the stress in the cup wall causes rupture at smaller drawing ratios. The effect is increased with larger tool diameters. The same observations are made for the edge radius of the die (Fig. 8*a* and *b*).

An increasing edge radius on the punch causes smaller stresses to a minimum point corresponding to an edge radius of about one-third of the punch diameter.[5] Larger edge radii give no absolute limit for the drawing ratio, but only a slight decrease. An upper limit for the edge radius on the die arises from the tendency for wrinkles to start when the blank-holder pressure reaches zero at an early stage in the drawing process (Fig. 9).

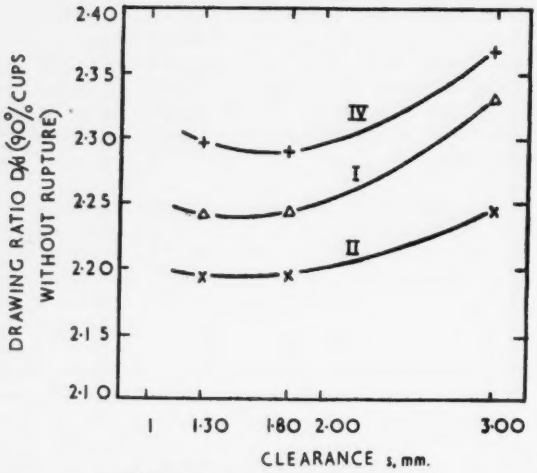
Tests showed that the edge radius has to be in a ratio of 1 : 4 to 1 : 6 to the tool dimension for cylindrical cups with diameters down to about 1 in. (25 mm.) and a minimum sheet thickness of 0·5–1·0 mm. With such values the classification of the five tested materials is possible within a large range of dimensions without any exceptional dispersion in the relation values for different tool sizes. In regard to the small slope of the curves shown in Fig. 8*b*, the tolerances of the edge figures only exert a secondary influence.

Drawing Speed—The influence of the drawing speed on the drawing-ratio values is of great interest in regard to variations in the practical working conditions. However, it has previously been shown by Swift,[4] and now in a special test series carried out by the Committee, that variations in linear speeds of the punch above about 0·12 in./sec. (3 mm./sec.) have no influence and can be neglected. The tests made by the Committee cover speeds of 0·2 and 6·06 in./sec. (5 and 154 mm./sec.) obtained in a hydraulic press and a screw press, respectively. Figures 10*a* and *b* show that as much as 30-fold variation in the speed has only a secondary influence upon the deep-drawing ratio, considering the statistical dispersion under normal testing conditions. The observation is identical for the five materials representing widely different physical and other properties. Different edge radii might give differences in strain hardening for different materials when the speed is varied, but no effect of that kind is to be found. There is also no reason to make calculations for other results for speeds between those used. At very low speeds, however, there is an increase in the drawing ratio, and the ratios for the different materials are expected to vary considerably. This is, of course, a normal effect of strain hardening in cold-working, corresponding



(*a*) d = 22·4 mm., r_g = 8 mm., s = 1·30 mm., = 1·00 mm. (*b*) d = 22·4 mm., r_g = 10 mm., s = 1·30 mm., t = 1·00 mm.

Fig. 10—Drawing ratio as a function of linear speed of punch



d = 32 mm., $r_s = r_d$ = 6 mm., t = 1·00 mm.

Fig. 11—Drawing ratio as a function of clearance between punch and die

to different true-stress/true-strain diagrams for different materials.

Clearance between Punch and Die—It is important to study the clearance between punch and die, as this determines the sheet thickness that can be tested in a certain tool. As the wall thickness of the cups increases towards the periphery of the specimen, a smaller clearance will cause a reduction in wall thickness and an elongation. The drawing force will then rise and a smaller drawing ratio may be expected. According to the different characteristics of the materials, variations in drawing properties might be expected, but in fact the pressure on the walls from the inside of the die and the outside of the punch permits a higher longitudinal stress in the wall before rupture occurs.[5] Thus a certain wall-thickness/clearance ratio will give a minimum drawing ratio. Larger clearances need smaller drawing forces as the contact pressure decreases.

Figure 11 confirms this theory. The dispersion of the drawing ratio in the diagram is different for small and large clearances. The minimum point is not greatly accentuated. The main observation is that the significance in correlation within the clearance figures used in the test is 100%.

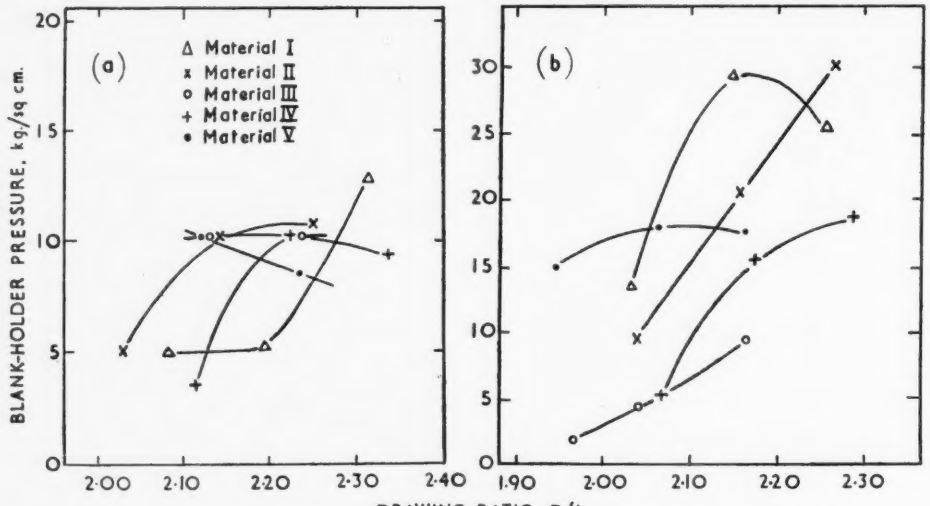
Consequently, it is proved that the same clearance can be used for varying sheet thicknesses within limits which may be narrow, when the nominal drawing ratio is required together with the relative size, or wide, when only the relation between the different materials is wanted. The Committee is at present working on the problem so as to provide recommendations for the limits mentioned.

Blank-Holder Pressure—In practice the blank-holder pressure is normally so great that wrinkles in the blank are completely avoided. The Committee has used the tendency for wrinkle formation as a criterion of the pressure necessary for different materials with different drawing properties. In general the following is observed.

The specific blank-holder pressure must be increased for greater drawing ratios and for greater tool diameters (Fig. 12*a* and *b*). The minimum pressure is not a significant figure for the drawability of the different materials, as the sequence, for instance, varies with different tool diameters.

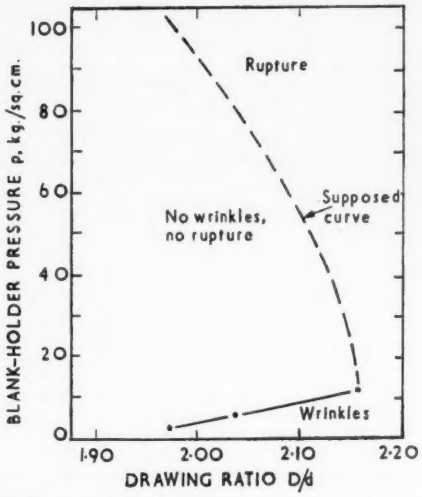
The possibilities of eliminating wrinkles also depend on the surface finish of the die, the blank holder, and the blank itself. The thickness tolerances of the sheet and the lubrication are also important. These factors give a statistical dispersion so wide as to spoil accuracy.

For test purposes the blank-holder pressure used for each material must be sufficient to eliminate wrinkles. Large plus tolerances have a secondary influence on the results. At a certain tool diameter an absolute maximum in the drawing ratio corresponds



(*a*) d = 22·4 mm., r_s = 8 mm., r_d = 10 mm., s = 1·30 mm., t = 1·00 mm. (*b*) d = 35 mm., r_s = 8 mm., r_d = 10 mm., s = 1·30 mm., t = 1·00 mm.

Fig. 12—Drawing ratio as a function of blank-holder pressure



d = 35 mm., r_s = 8 mm., r_d = 10 mm., s = 1·30 mm., t = 1·00 mm.

Fig. 13—Drawing ratio as a function of blank-holder pressure: material V

to a certain blank-holder pressure (Fig. 13). An attempt was made to find the pressure which results in rupture at a drawing ratio only a few per cent. below the absolute maximum. This pressure is, however, so great that there is no risk of attaining it under normal conditions.

Surface Finish—The surface finish of the die and blank holder is so important for the test results that special care must be taken in the grinding and polishing operations during toolmaking and in regard to wear on the tool during use. The testing tools should have a definite surface finish, determined by r.m.s. or H values.* The wear consists chiefly of pick-up marks, which are especially intensive with materials which have a tendency to ' weld ' to the tool at high pressure (Fig. 14).

Lubrication—This is of even greater importance than surface finish. Special test series have been made to classify the different lubrication fluids, and a wide quality range exists from a functional point of view. Every material needs its special lubricant to give the highest value for its maximum drawing relation. Nevertheless, a classification can be made of the five materials chosen for the Committee's tests without difficulty, even when using the same lubricant, although naturally the ideal maximum drawing ratio is not attained in this way.

CONCLUSIONS

Owing to its complicated nature, deep-drawing with material sliding needs a test which approximates in all respects to the deep-drawing process. Most of the test properties have to be fixed with great accuracy.

Deep-drawing with pure elongation is easier to test. A modification of the Erichsen test is quite satisfactory. The test properties must be fixed with great accuracy, and the criterion for drawing quality must be the pressing force at the point of rupture, instead of the depth of rupture, as measured in the normal subjective way.

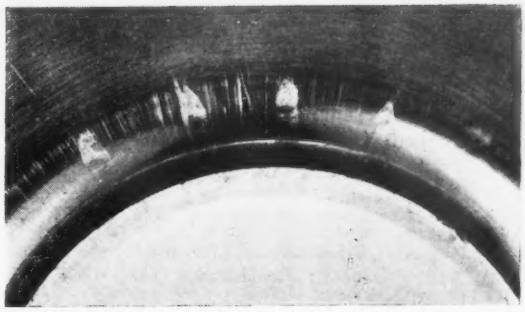


Fig. 14—Pick-up marks on die edge resulting from tests with stainless steel (material III) × 3

In view of the results obtained, the Committee intends to propose the two deep-drawing tests described. The possibility of classifying materials with small variations in physical properties is at present being investigated at Sandviken. The test methods will shortly be presented to corresponding organizations in other countries in an attempt to arrive at an international standard for testing the drawing qualities of sheet.

* r.m.s. = Surface finish defined by the root mean square of the peak-to-valley distances of irregularities, and H values are simple arithmetic means of these distances.

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Calculation of Roll Pressure and Energy Consumption in Hot-Rolling

By Gunnar Wallquist

SYNOPSIS

The paper shows that the calculation of roll pressure and energy consumption in hot-rolling may be based either on a mathematical analysis of the deformation or on empirical values obtained experimentally during rolling, the latter method being the only reliable one at present. Previous work in the field is reviewed. The present investigation includes studies on the deformation process in hot-rolling and the determination of numerical values for coefficients necessary for the calculations. The influence of steel composition, temperature, reduction, and material thickness on roll pressure, energy consumption, spread, and forward slip was studied on 16 steels. Tests were made in the new experimental mill at the Royal Institute of Technology, Stockholm. General methods for calculating roll pressure and energy consumption are described. Besides the specific roll pressure, the roll-pressure coefficient, previously used in cold-rolling calculations, is introduced. Part of the results are summarized in diagrams showing the specific roll pressure, the roll-pressure coefficient, and the torque-arm coefficient as functions of various rolling factors. The experimental results in their entirety will be reported later. 931

Introduction

In assessing electrical and mechanical equipment and calibrating rolls when erecting new rolling mills or when rebuilding or improving old ones, it is important to know the stresses arising in different parts of the equipment, and also the energy needed to carry out the rolling process. Primary data are the roll pressure and the torque. Numerical values for these factors, applicable to different types of rolling, may be obtained by calculations, based either on a mathematical analysis of the deformation process or on data obtained by measuring the roll pressure and energy consumption in rolling mills in operation. Purely theoretical considerations have not yet led to an entirely reliable method for this calculation. In assessing a particular rolling operation it is therefore most convenient to use constant relative values, previously obtained by measurement. Simultaneous study of the deformation process and determination of constants needed for making the calculations is therefore an important research task within the rolling technique. For such investigations industrial rolling mills as well as experimental rolling mills specially designed for the purpose have been used.

Earlier investigations were only concerned with determining the energy consumption. The first experiments were probably those of Boeck,[2] who studied a rolling-mill steam engine as early as 1873, and of Blass and Daelen[1] who, seven years later, also studied steam engines. In 1906 Puppe, supported by the Verein deutscher Eisenhüttenleute, started the first comprehensive investigation of power consumption.

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Table I

COMPOSITIONS OF STEELS INVESTIGATED

Steel No.	Composition, %									
	C	Si	Mn	P	S	Cr	Ni	W	Mo	V
1	0·10		0·47	0·063	0·026					
2	1·03	0·22	0·27	0·030	0·024					
3	1·02	0·24	0·35	0·027	0·016	0·56				
4	1·00	0·30	0·26	0·029	0·013	1·19				
5	0·99	0·36	0·33	0·026	0·022	1·84				
6	0·13	0·25	0·55	0·020	0·018	0·75	3·00			
7	0·55	0·26	0·46	0·017	0·013	0·95	2·94		0·31	
8	1·28	0·20	0·35	0·024	0·010	0·17				
9	0·09	0·24	0·32	0·016	0·025	13·3				
10	0·12	0·31	0·31	0·016	0·014	16·8				
11	0·22	0·80	0·50	0·020	0·015	24·0	0·30			
12	0·08	0·45	0·67	0·015	0·015	18·2	9·9		0·60	
13	0·10	0·50	0·40	0·016	0·017	16·7	20·6			
14	0·34	0·18	0·46	0·017	0·015	14·3	0·22	1·18		
15	2·01	0·38	0·72	0·020	0·020	13·5				0·20
16	0·74	0·25	0·39	0·032	0·011	4·65		17·8	0·44	1·12

The results were published partly in book form (1909[15] and 1910[16, 18]), partly in a paper.[20] Similar investigations, also performed at steelworks, were made by Smitz,[29] Hopper,[10] Werth,[42] Waldorf,[36] Weyel,[43] the present author,[37, 38] and others.

The need of constants for different calculations soon caused the investigations to be extended to include the determination of roll pressures as well. The work was continued at steel mills but, to facilitate the research, experimental rolling mills were soon built at different institutes for higher technical education.

Among investigations at steel mills, including determination of both roll pressure and energy consumption, may be mentioned those of Puppe,[17, 19, 21] Hoff and Dahl,[7] and the present author in collaboration with Leufvén and Stjernquist[39] and Leufvén and Ollén.[40]

Researches carried out in experimental mills include those of Siebel,[25] Siebel and Fangmeier,[27] Pomp and Weddige,[14] Emicke and Benad,[5] Pomp and Lueg,[13] and others.

These investigations have shown that several factors influence roll pressure, energy consumption, spread, and forward slip in hot-rolling. The most important of these factors, the so-called 'rolling factors', are the steel properties (influenced by composition, temperature, or other causes), the reduction, the method of deformation, and the diameter, surface properties, lubrication, and cooling of the rolls. Previous investigations have only elucidated the numerical importance of the different factors to a limited extent. Many attempts have been made to analyse the deformation mathematically and to deduce generally applicable formulæ for the calculation of both roll pressure and energy consumption. Some of the purely theoretical analyses of the deformation during rolling such as those by Siebel,[23-26] Trinks,[31-33] von Kármán,[11] Hoff and Dahl,[8] Orowan,[12] and Tselikov,[34] are still of considerable interest, but could not be practically applied in hot-rolling. On the other hand, the method given by Ekelund[3, 4] and intended for practical calculations has been widely recognized for a number of years and is probably the method most generally used today. Because of insufficient experimental foundations, however, the use of the method is limited to certain types of rolling; for others it gives completely erroneous results.

As a further step towards solving the problem of calculation it has for a long time been imperative to clarify the influence of the different rolling factors on roll pressure, energy consumption, and material deformation by means of extensive and careful investigations of the hot-rolling process. In 1951, therefore, the author started a series of such investigations in an experimental mill newly installed in the Institution for Working and Treating of Steel at the Royal Institute of Technology, Stockholm. The first part of these investigations ended early this year (1954) and the present paper is the first communication on results obtained.

STEELS INVESTIGATED

The steels were delivered in rolled condition as flat sections 50 mm. wide × 2·5, 5, 10, and 20 mm. thick. They were delivered sheared in suitable lengths between 1000 and 1800 mm. The analyses of the steels are given in Table I. All the steels were air-cooled after hot rolling, except the martensitic steels 15 and 16, which had been slowly cooled.

METHOD OF INVESTIGATION

The test bars were heated individually in an electrical heating furnace, situated immediately in front of the stand, so that the bar could remain partly in the furnace during the rolling of the entering end. The heating time was adjusted according to quality and dimensions. To obtain a uniform surface with a thin oxide layer, pure nitrogen gas was introduced into the furnace from a container. Each test comprised one pass. Chilled cast-iron rolls with a diameter of 340 mm. were used. To prevent oxidation after rolling, the test bars were quenched by water spray immediately after the pass. The quantity of

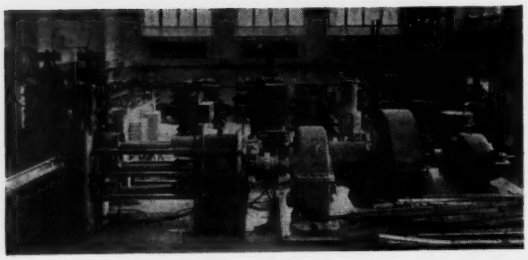


Fig. 1—Experimental rolling mill at the Royal Institute of Technology, Stockholm

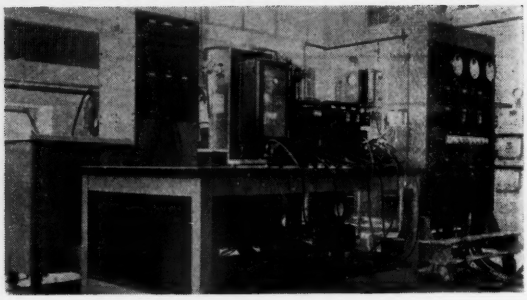
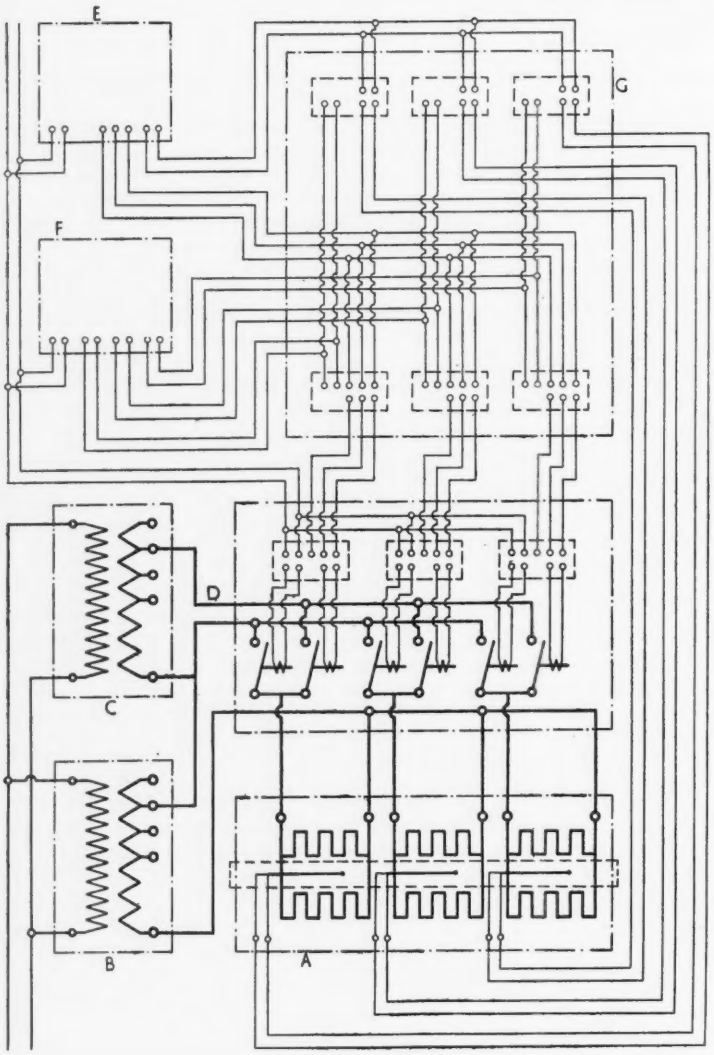


Fig. 3—Temperature regulating device of experimental mill



A. Electric furnace
B. Main transformer
C. Transformer for periodical voltage addition
D. Panel with contactors and contactor relays
E. Regulating potentiometer
F. Rotating switch
G. Panel with periodical working relays for thermocouples and contactor relays

Fig. 2—Wiring diagram for furnace temperature regulation

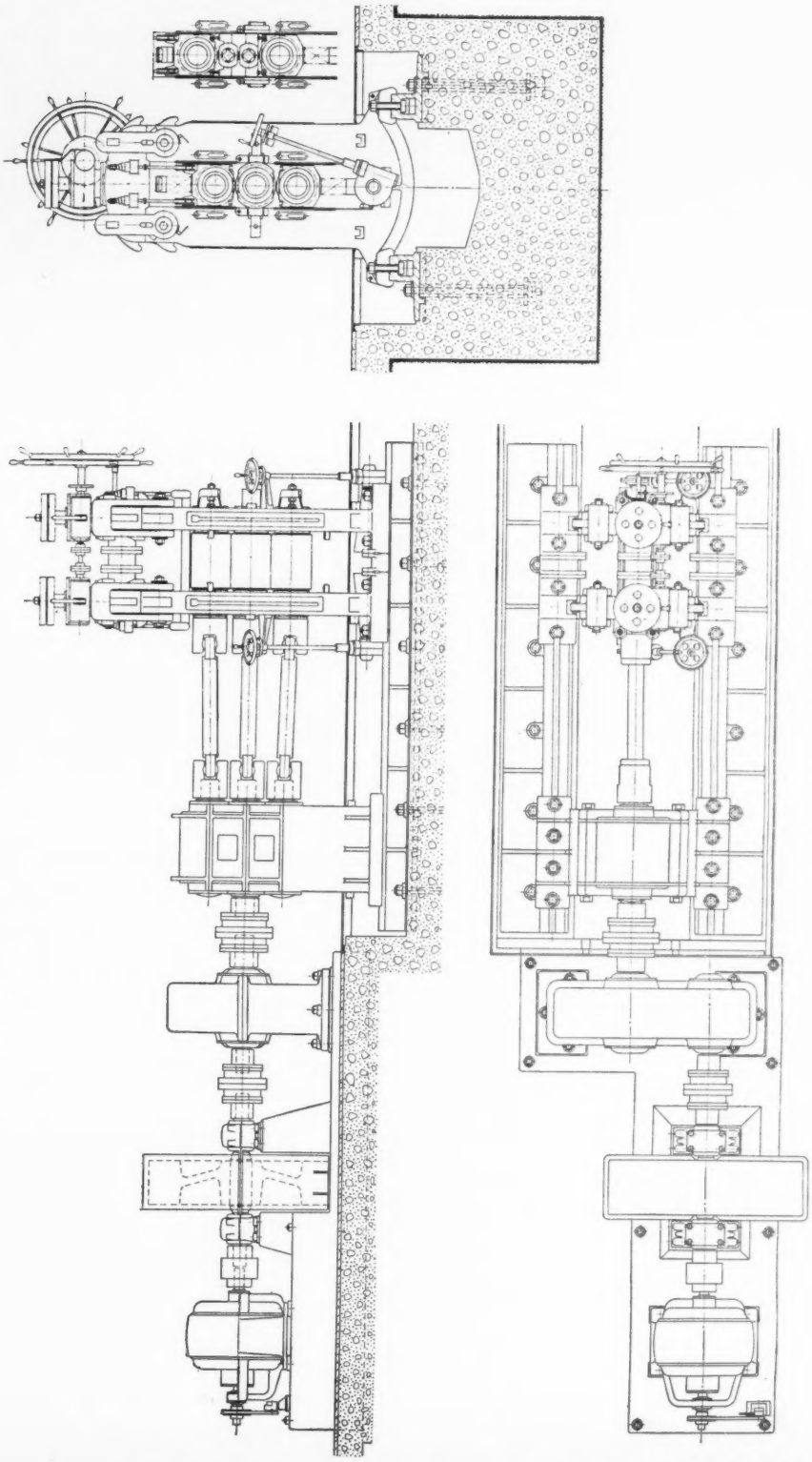
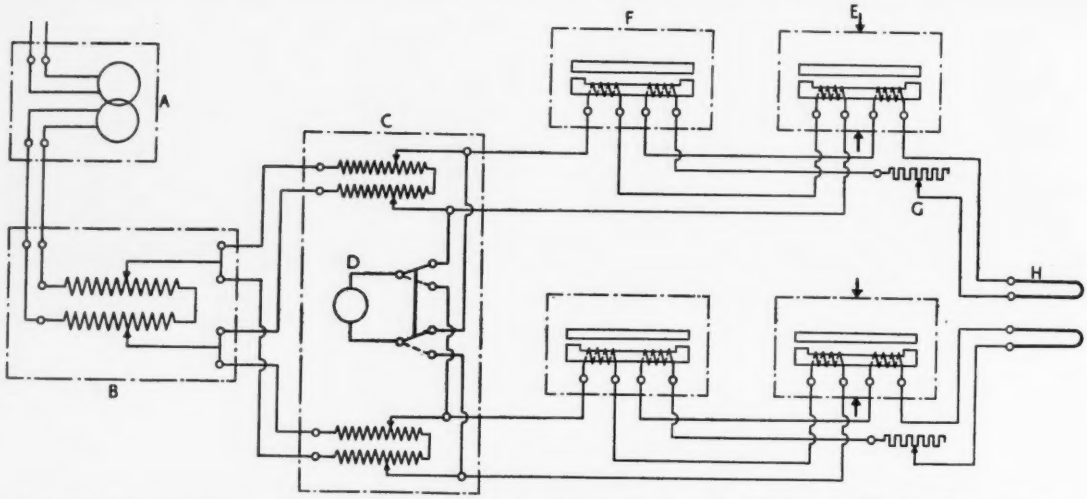


Fig. 4—Experimental rolling mill



A. Constant voltage transformer *B.* Main transformer *C.* Voltage regulation transformers *D.* Voltmeter *E.* Induction roll-pressure gauges *F.* Comparison sets *G.* Resistances *H.* Oscillograph galvanometers

Fig. 5—Wiring diagram for roll-pressure measurement

water was adjusted so that hardening of the harder steel qualities was avoided.

Double test studies were made on the influence of temperature, reduction, and height of material on total and specific roll pressure, energy and power consumption, spread, and forward slip. The temperature factor was studied by rolling all materials at 800°, 900°, 1000°, and 1100° C., and by several extra tests, designed to study the properties of the steels in the transformation range. Different test series were made with linear reductions of 10%, 20%, 30%, and 40% on all the initial thicknesses, *i.e.*, 2·5, 5, 10, and 20 mm. All the tests were made with an average roll speed of 100 r.p.m., corresponding to a peripheral speed of 1·78 m./sec. The total roll pressure was measured and registered by a device described in the following section. The total energy consumption is the sum of the energy quantities delivered by the rolling-mill motor and the flywheel.

The steel temperature in the furnace was measured with a Ni/Ni–Cr thermocouple and with an optical pyrometer.

EQUIPMENT USED

Electric Furnace

A resistance furnace with Kanthal wire elements was used ; it was divided in three electrically independent sections. The muffle consisted of a welded rectangular tube of heat-resisting steel with interior dimensions 150 mm. × 80 mm. The ends had gas-tight shutters, and small tubes were attached for introducing the nitrogen gas and for measuring the temperature.

Figure 1 shows the experimental mill and the furnace. Figure 2 is a wiring diagram for the temperature regulation of the furnace, and Fig. 3 shows the regulating device. Each section is an independent unit. The primary impulses were obtained from a temperature regulator, a relay panel and a contactor panel being used for regulating the effect. By the combined action of a rotating switch device and a relay panel, the thermocouples in each furnace are connected in turn to the regulator, from which the regulating impulses simultaneously proceed *via* the relay panel to the two contactors of the section. The resistance elements were fed from a single-phase main transformer for secondary voltages between 27 and 70 V. and an additional transformer in series with the first of 0–12 V. The power regulation was arranged as a combined time–voltage regulation by connecting either the main transformer voltage only or this voltage plus that of the additional transformer, with double contactors.

Experimental Rolling Mill

The rolling mill consists of a stand, a pinion gear, a main gear, a free-carried rapid flywheel, and a D.C. motor fed in Ward–Leonard coupling from a motor generator. The stand can be assembled as 2-high, 3-high, or 4-high. Arranged 2-high or 3-high, roll diameters of 250–500 mm. are possible ; the 4-high assembly can be used with working rolls of 125–175 mm. dia. and back-up rolls of 330–350 mm. dia. The roll-body length may be either 450 or 600 mm. The middle roll is fixed, the top roll is adjusted by screw-down with worm gear, and the bottom roll by a wedge device with worm gear. During the tests the stand

Fig. 6—Equipment for roll-pressure measurement

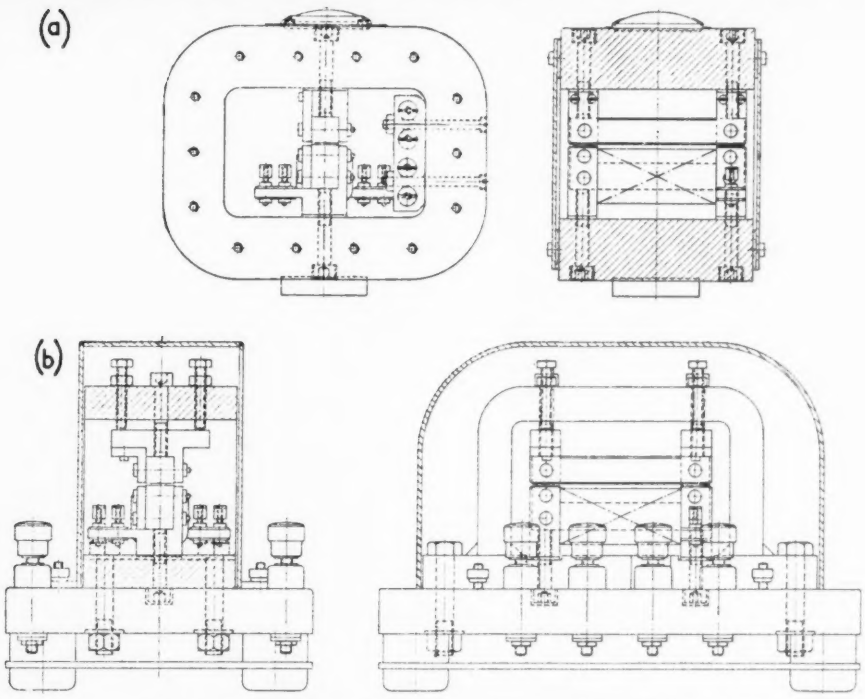


Fig. 7—(*a*) Induction roll-pressure gauge; (*b*) comparison set

was arranged 2-high. Figure 4 shows the mill assembled 3-high and 4-high.

The power required during the pass is obtained partly from the motor and partly from the flywheel. The motor has a maximum continuous effect of 100 h.p., and its speed can be regulated between 0 and 1100 r.p.m., corresponding to 0–382 r.p.m. at the stand. The train has an energy coefficient reduced to the main axle of 38,900 kg. sq. m.

Equipment for Roll-Pressure Measurement

Roll-pressure gauges of induction type, designed by the author, were used in the tests. They were placed between the screwdown and the top roll bearing. The two roll-neck pressures were registered by a three-loop oscillograph. A zero voltage method was employed, the gauges being balanced against identical counter-connected comparison sets.

Figure 5 is a wiring diagram of the measuring device, Fig. 6 is a photograph of the equipment, and Fig. 7 shows the induction gauges and the comparison sets. The former contain an elastic rectangular steel frame, influenced by the roll-neck pressure, and two iron cores of transformer sheet. One core has a primary and a secondary coil, and the second serves as a yoke. The primary circuit is fed with A.C. (5 V., 50 cycles). The voltage in the secondary coil depends on the primary voltage and on the air gap between the two cores. If the roll pressure causes a compression of the steel frame the air gap decreases, the induction is improved, and the voltage in the secondary circuit rises. The relation between secondary voltage and pressure is almost linear at constant primary voltage. The comparison sets are, in principle, identical to the pressure gauges, but they have an adjustable air gap which is kept constant during measuring. The A.C. was obtained from a constant-voltage set and a transformer. For adjusting the primary voltage there were two smaller transformers in each gauge regulated by a voltmeter. Adjustment of the diagram size in the oscillograph was located in the secondary circuit, which for this reason was supplied with a voltage-dividing universal rheostat.

Figure 8 shows an oscillogram for a pass. The paper speed is 10 mm./sec. The entering sine curves represent the circuit voltage before the pass. The big deflections show the change in voltage during the pass, and the difference in voltage gives the measured roll-neck pressures.

The pressure-measuring device was calibrated at regular intervals in a hydraulic press against a mechanical pressure meter.

Equipment for Energy Measurement

The energy required during the pass is delivered partly by the mill motor and partly by the flywheel. The motor power is the product of the amperage, the voltage, and the efficiency. The voltage was kept constant during the pass. The current was measured with the third loop of the oscillograph and was registered on the oscillogram shown in Fig. 8. The efficiency was known from the testing of the electrical equipment. The energy delivered by the flywheel was obtained from a speed/time diagram taken up with a registering voltmeter with suppressed zero,

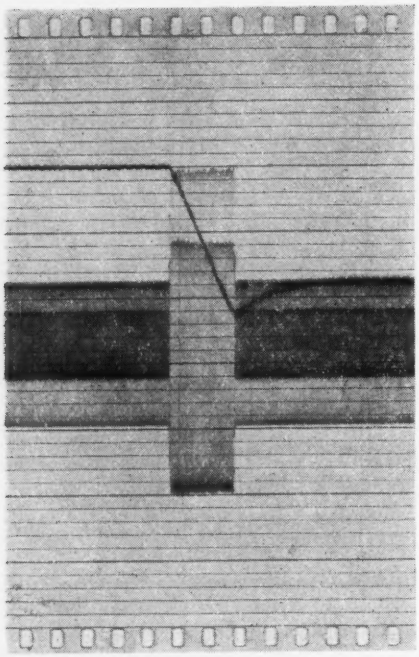


Fig. 8—Oscillogram for the two pressure gauges and the current intensity to the rolling-mill motor

which was fed from a D.C. generator with permanent magnets connected to the train.

CALCULATING METHODS

Roll Pressure

As already mentioned, the attempts made by different investigators to analyse the deformation in the area of contact and to deduce formulæ for calculating the varying specific pressure in this area as well as the total roll pressure have not led to satisfactory results. The most reliable method of calculation so far is to start either from the mean value of the specific roll pressure and the projection of the area of contact on a plane parallel to the direction of rolling or, as in cold-rolling,[30] from a roll-pressure coefficient and the product of roll diameter and mean width of the material.

The specific roll pressure and the roll-pressure coefficient may be obtained directly from available diagrams, representing normal rolling operations. Another possibility is to use standard diagrams, corrected by coefficients for the influence of different rolling factors.

In using the specific roll pressure the following equation is valid for rectangular sections:

$$F_v = p_{vm} \times b_m \times x_0 \quad \text{.....(1)}$$

where F_v = total roll pressure, kg.
p_{vm} = mean specific pressure, kg./sq. mm.
b_m = mean width of material during pass, mm.
x_0 = projection of the area of contact on a plane parallel to the direction of rolling.

The value of x_0 can be calculated with sufficient accuracy from the equation

$$x_0 = \sqrt{\left(D \times \frac{h_f - h_a}{2}\right)} = \sqrt{\left(D \times \frac{c}{100}\right)} \quad \text{.....(2)}$$

where D = roll dia., mm.
h_f, h_a = material thickness before and after pass, respectively, mm.
c = relative linear compression, %.

Starting from a roll-pressure coefficient, the following equation can be used :

$$F_v = k_v \times b_m \times D \quad \text{.....(3)}$$

where k_v is the roll-pressure coefficient in kg./sq. mm. and is valid per mm. of bar width and roll diameter.

Energy Consumption

To keep the mill running idle, a certain idle power is required which represents, during a certain time, an idle work (W_t). During the pass the work necessary for deformation, the rolling work (W_v), is added. The latter consists of the work of deformation required to cover the internal friction in the material rolled (W_{vd}), the external work of friction due to the friction between the material and the rolls (W_{vf}), and the train friction work caused by the increased friction in the mill machinery (W_{vt}), such as roll bearings, gears and gear bearings, clutches, etc. These may be expressed as:

$$W = W_t + W_v \quad \text{.....(4)}$$

$$W_v = W_{vd} + W_{vf} + W_{vt} \quad \text{.....(5)}$$

where W is the total work and all are expressed in kg. m., h.p.-sec., kW.sec., or kWh.

If the torques (in kg. m.) are substituted for these works, the two equations will be :

$$M = M_t + M_v \quad \text{.....(6)}$$

$$M_v = M_{vd} + M_{vf} + M_{vt} \quad \text{.....(7)}$$

The idle torque M_t can be calculated in the ordinary way from the known train data.

The torque of deformation M_{vd} and the torque of friction between the material and the rolls M_{vf} cannot be distinguished by measuring. They can be calculated from the following equation:

$$M_{vd} + M_{vf} = 2F_v \times x \quad \text{.....(8)}$$

where x is the arm of torque in mm. of the total roll pressure. It can be expressed in relation to x_0 as

$$x = k_x \times x_0 \quad \text{.....(9)}$$

where k_x is the so-called torque-arm coefficient.

The main part of the train friction work is located at the roll bearing of the stand. This part of the torque may be written:

$$M'_{vt} = F_v \times \mu_t \times d_v \quad \text{.....(10)}$$

where μ_t = coefficient of friction for the roll bearings
d_v = effective roll-neck dia., mm.

For spherical roller-bearings the value of the friction coefficient may generally be assumed to be 0·002, and the effective neck diameter may be measured to the centre of the rolls in the bearings.

The other part of the train friction torque can either be calculated for the different parts of the machinery separately or can be obtained by aid of the total efficiency of these components. In the latter case the friction torque is

$$M''_{vt} = (1 - \eta_t)M_v \quad \text{.....(11)}$$

where η_t is the efficiency of all movable train parts

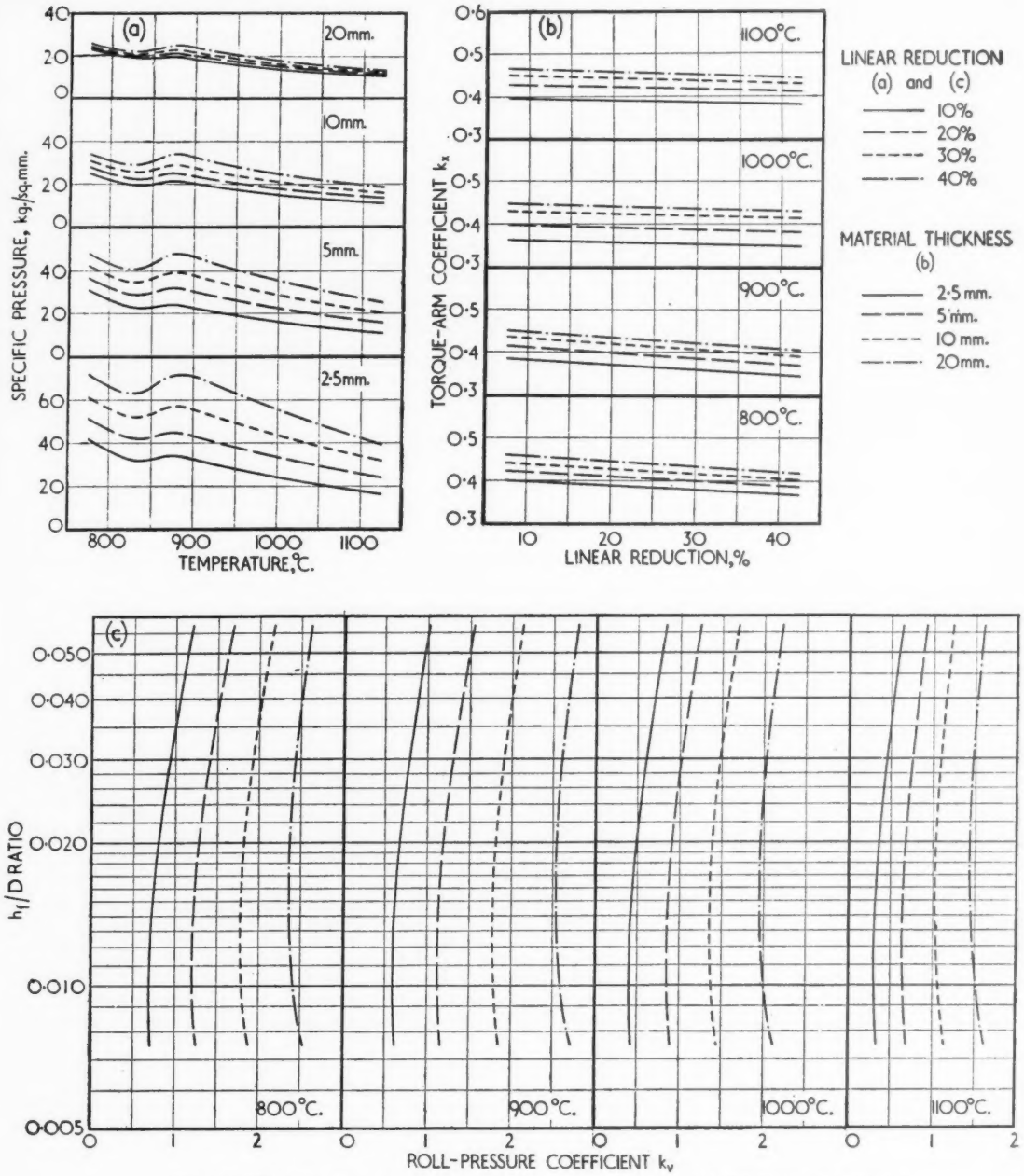


Fig. 9—Effect of steel temperature, reduction, and material thickness for steel 1

between the motor and the stand. The rolling torque is then

$$M_v = \frac{F_v}{\eta t} \times (2k_x \times x_0 + \mu_t \times d_v)$$

$$= \frac{k_v \times b_m \times D}{\eta t} \times \left[2k_x \times \sqrt{\left(D \times \frac{c}{100}\right)} + \mu_t \times d_v\right] \quad ..(12)$$

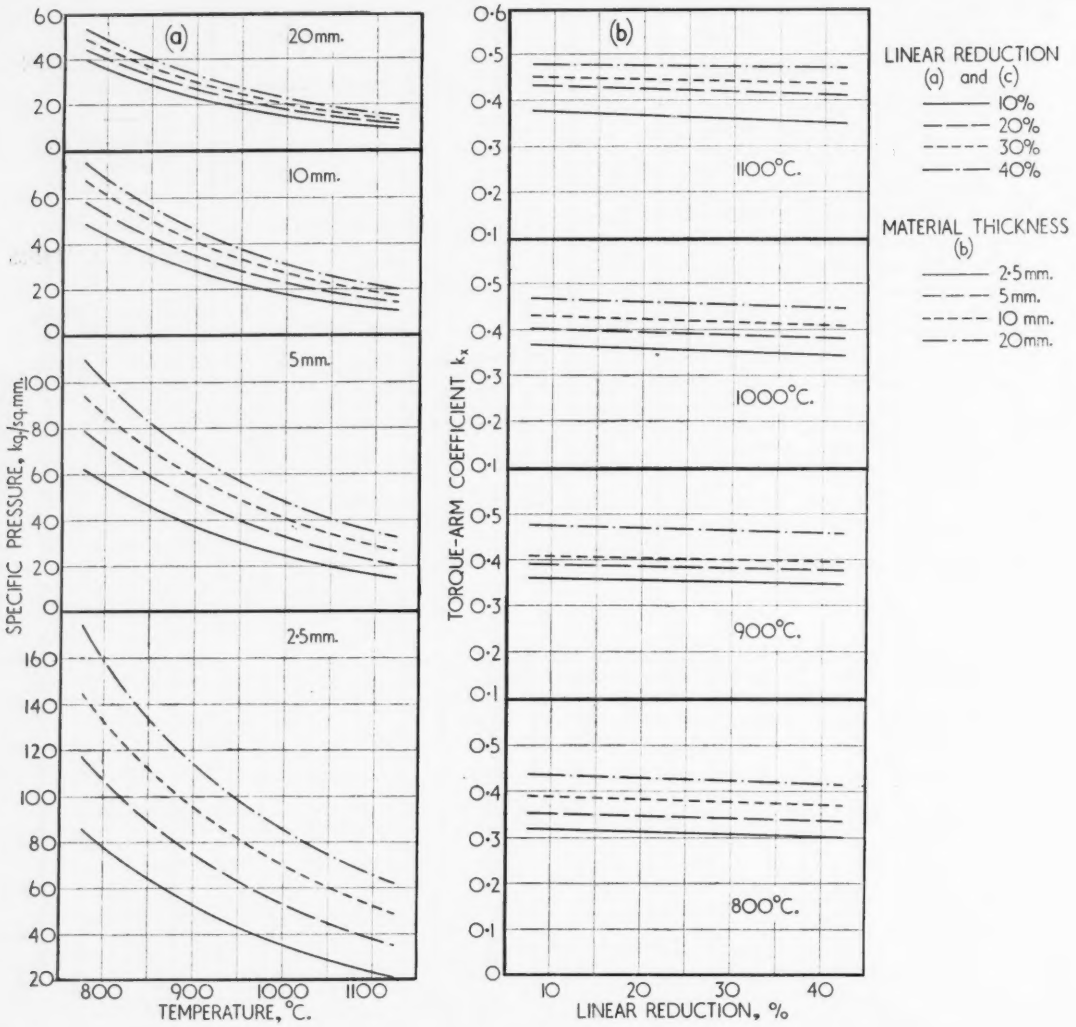
and the rolling work is

$$W_v = \frac{\pi \times n}{30} \times M_v = \omega \times M_v \quad ..... (13)$$

where n = mean roll speed, r.p.m.
ω = corresponding mean value, radians/sec.

Numerical Values of the Coefficients

In addition to the study of the behaviour of the different steels in deformation, numerical values were determined for p_{rm}, k_v, and k_x at varying temperatures, reductions, and material thicknesses. These values are limited to the rolling of rectangular sections in a certain range of dimensions, but they also give



hitherto unknown indications for determining numerical values outside this range. The author's research programme also includes plans for studying the influence on the numerical values of the coefficients of width and form of the material, roll diameter, rolling speed, roll material and surface, and lubrication and cooling of the roll surface.

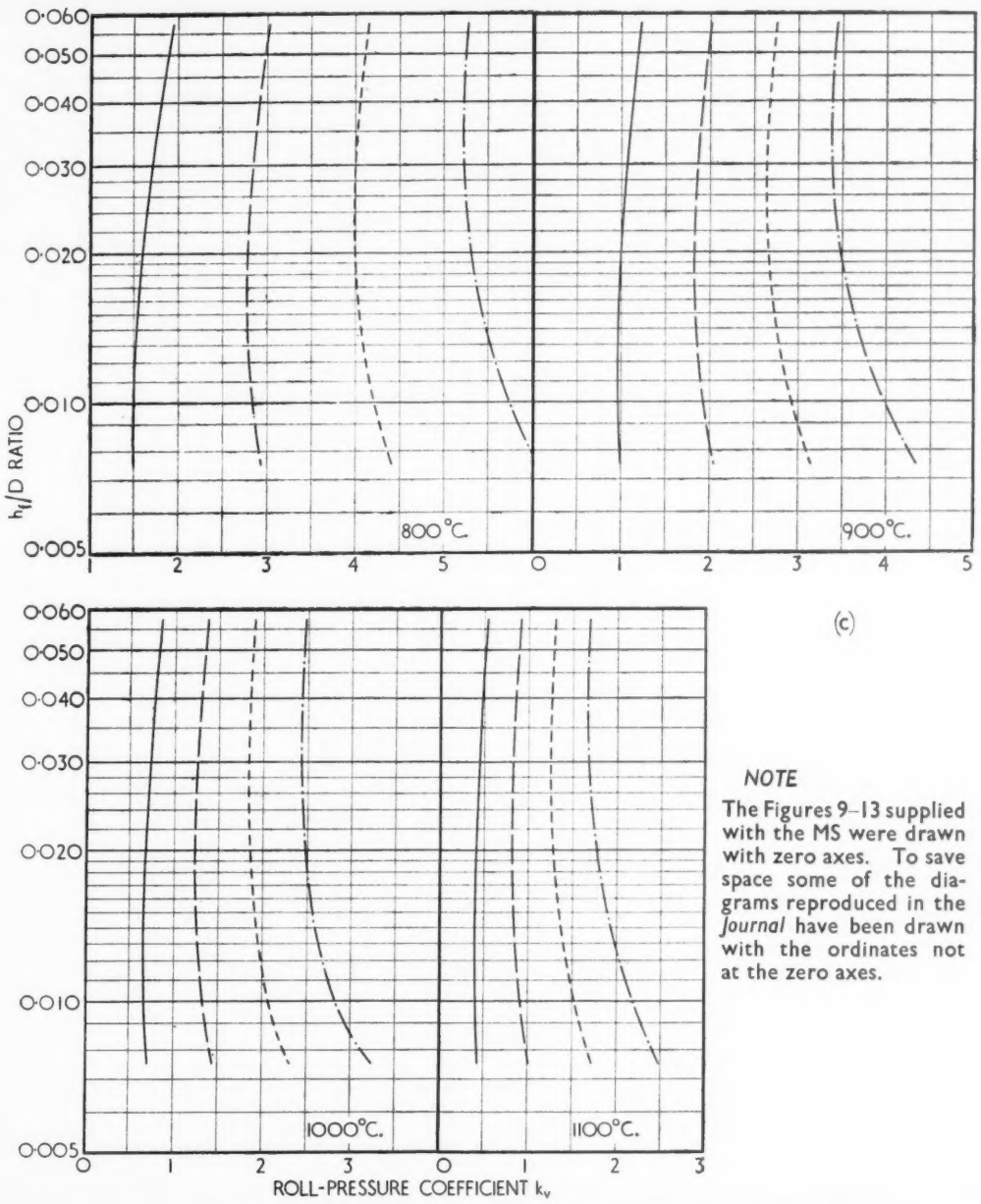
RESULTS OF PRESENT INVESTIGATION

Introduction

Previous investigations on the influence of different factors on roll pressure and energy consumption in hot-rolling have been limited to one or a few steel qualities. The only more comprehensive work is that by Pomp and Lueg,[13] which is an account of the tests made in the small experimental mill at the Kaiser-Wilhelm Institut in Düsseldorf in 1930–1939. This account shows that at low temperatures soft carbon steel has the lowest specific roll pressure and that this increases with carbon content. At medium temperatures, however, the difference between the various carbon steels was found to be small, and at high temperatures hard carbon steels are more easily worked than soft ones. Low-alloy steels behave like carbon steels with the same carbon content, but the high-alloy martensitic and austenitic steels require a much higher roll pressure, especially in the presence of certain carbides. Generally speaking, increase in reduction and decrease in material thickness cause an increase in specific roll pressure.

Most other investigations in this field are accounted for in two recently published books, one by Underwood[35] and the other by Hoff and Dahl.[9]

The results obtained in the present investigation have been brought together in diagrams, showing for each of the 16 steels the variation of the numerical values of total and specific roll pressure, roll-pressure coefficient, rolling power, torque-arm coefficient, coefficient of spread, and forward slip with temperature, reduction, and material thickness. The present



(c)

Fig. 10—Effect of steel temperature, reduction, and material thickness for steel 2

paper gives only a brief account, limited mainly to five of the steels investigated and to the coefficients which, according to the methods of calculation mentioned, are necessary for the determination of total roll pressure and energy consumption during a pass. A complete account of the results obtained in the present investigation will be published later as a special research report.

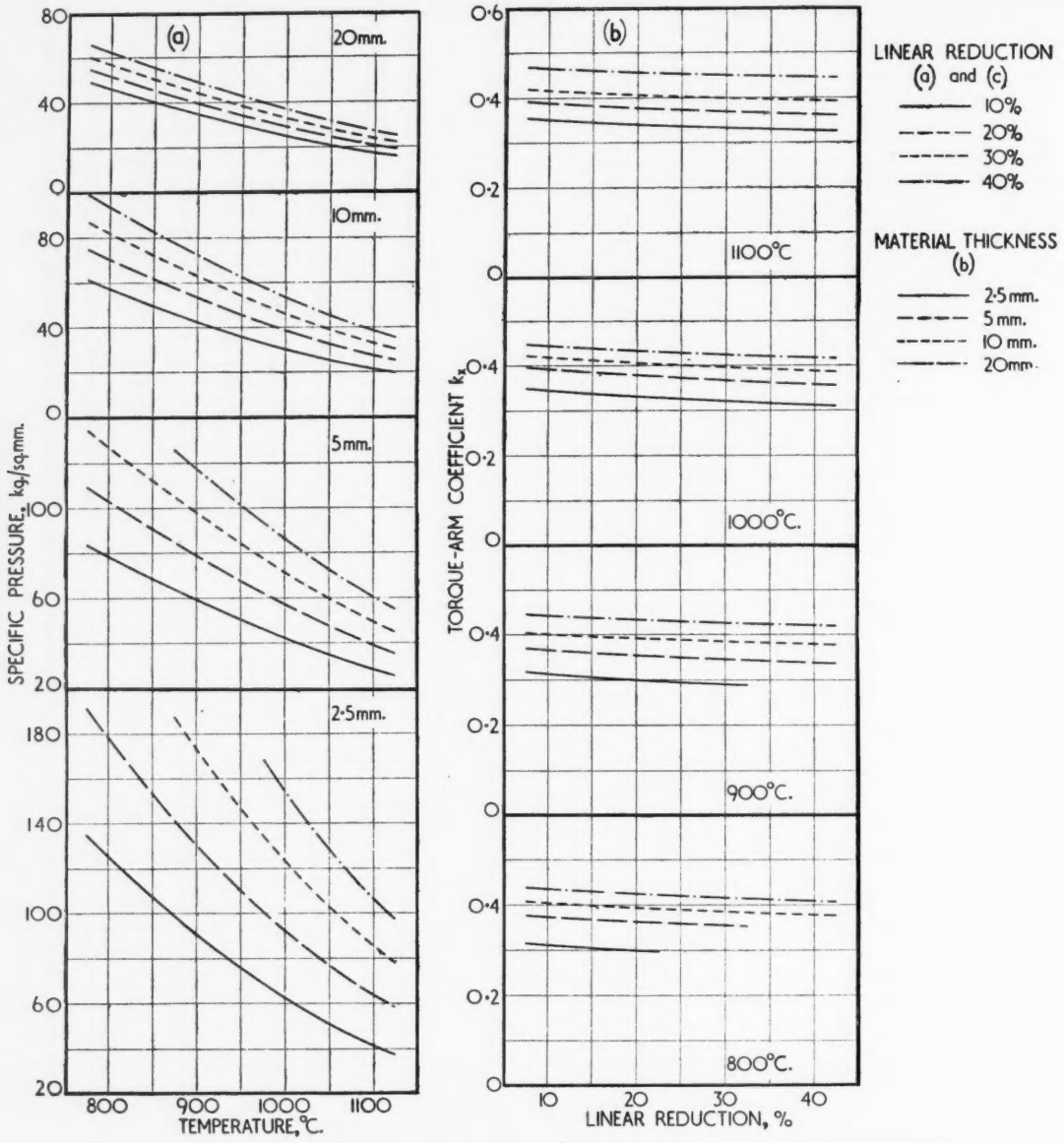
Specific Roll Pressure

The observations on the effect of structural changes, steel composition, steel temperature, reduction, and material thickness for steels 1, 2, 12, 15, and 16 are shown in Figs. 9–13, and are briefly discussed below.

Structural Changes—A transformation during rolling was, as expected, found to have a notable influence on the roll pressure. For steels 1, 9, 14, 15, and 16 the transformation to austenite occurs in the temperature range investigated. This causes a rise in the deformation resistance, in certain cases (*e.g.*, steel 14) of more than 50%. A larger reduction at constant roll pressure will be obtained at higher

NOTE

The Figures 9–13 supplied with the MS were drawn with zero axes. To save space some of the diagrams reproduced in the *Journal* have been drawn with the ordinates not at the zero axes.



working temperatures when rolling within the range than at lower temperatures. The face-centred lattice shows a greater resistance to deformation than the body-centred. Similar observations have been made in rolling tests by Siebel[25] and by Siebel and Fangmeier,[27] in compression tests by Riedel[22] and by Hennecke,[6] and in impression tests by the present author.[41]

Steel Composition and Temperature—The composition and temperature of the steel are intimately interdependent factors. Generally speaking, the roll pressure increases with rising carbon content and other alloy contents as well as with falling temperature, but very differently in different cases.

Steel 1, a soft carbon steel, is, depending on reduction and material thickness, 1·6–2·2 times as hard at 800° as at 1100° C.; for steel 2, a hard carbon steel, the corresponding figures are 2·4–4·5 times. The difference between the two steels is thus large at lower temperatures but slight at 1100° C. As found in the investigations at Düsseldorf, at 1200° C. the roll pressure is probably lower for the hard steel. This phenomenon is closely related to the lower melting point of the latter. The ratio between the roll pressure for steels 1 and 12 can be seen from Figs. 9*a* and 11*a*. A minimum value of about 1·7 is obtained in rolling thick bars with small reductions at 1100° C., and a maximum value of about 4·2 at rolling thin

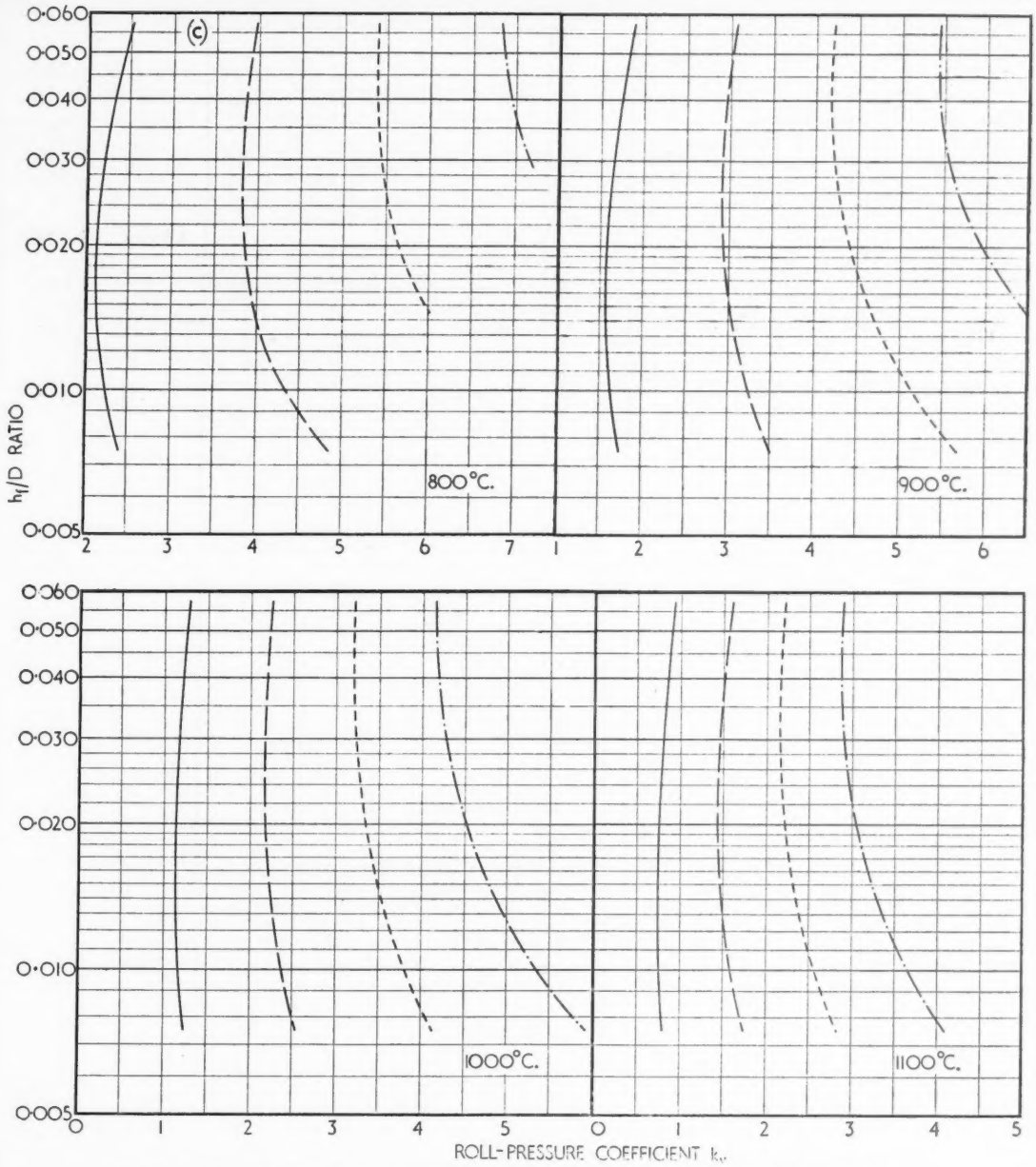
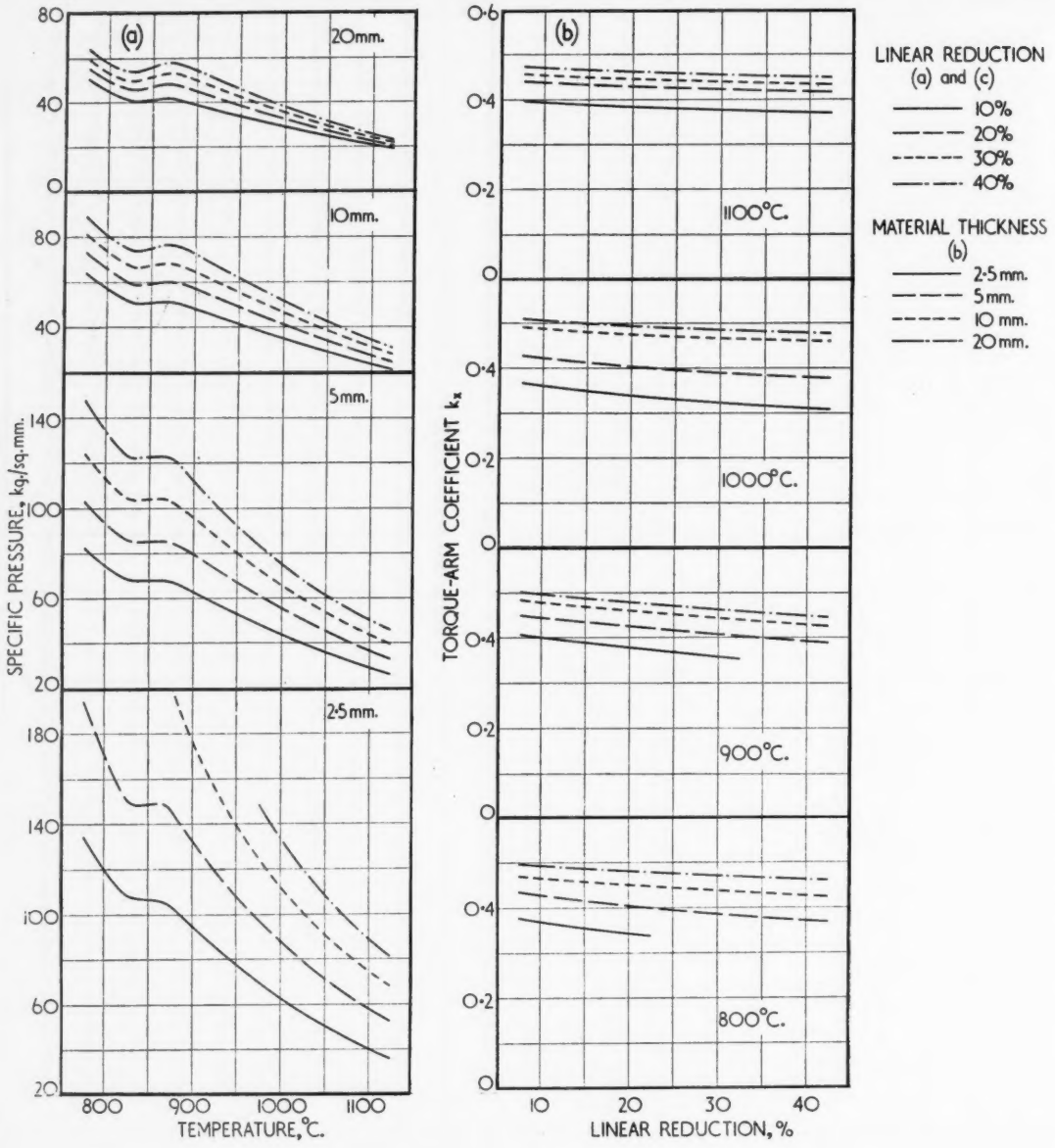


Fig. 11—Effect of steel temperature, reduction, and material thickness for steel 12

dimensions with high reductions at 800° C., a temperature below the effective transformation temperature of the carbon steel. These variations should explain why opinions on this point have diverged so much in practice : they were based on measurements made under widely differing conditions.

A rise in alloy content at constant carbon content may increase or decrease the roll pressure. For low-alloy steels the change is insignificant. The three chromium steels 3, 4, and 5 show at low temperature a slight increase in resistance to deformation with rising chromium content, but at high temperature there is a small decrease. The heat-resisting steels 10 and 11 show on the whole the same tendency, and the amount of reduction seems to be important at low rolling temperatures. If the alloy content is still more increased by substituting nickel for part of the iron, as in the austenitic heat-resisting steels 12 and 13, the roll pressure decreases with rising alloy content at low as well as at high temperature.

The highest roll pressures are obtained for steels with medium or high carbon contents and with high



alloy content: the stainless knife steel 14 and the cold-working steel 15 are examples of this. If the alloy content is still higher, as in the high-speed steel 16, a lowering of the deformation resistance is obtained, caused by the large alloy content.

Reduction—In the early rolling investigations made by Siebel[25] in 1930 at the Kaiser-Wilhelm Institut it was shown that not only the total but also the specific roll pressure increases with the reduction. The present author has established how this relationship is influenced by steel composition, temperature, and material thickness. This can be studied in detail in Figs. 9*a*, 10*a*, 11*a*, 12*a*, and 13*a*.

For the soft carbon steel 1 an increased linear reduction from 10% to 40% causes, depending on the material thickness, a rise in the specific roll pressure at 1100° C. of about 17–30%, but at 800° C. it is about 80–140%. Still greater differences have been ascertained: for steel 10 there is an increase of up to 220%. This dependence on temperature and material thickness has not previously been sufficiently observed.

For small thickness, *i.e.*, a low h_f/D ratio, the flattening of the rolls also has an effect. So long as the pressure distribution in the area of contact is not known, it is difficult to determine this deformation by calculation. Research in this field meets with

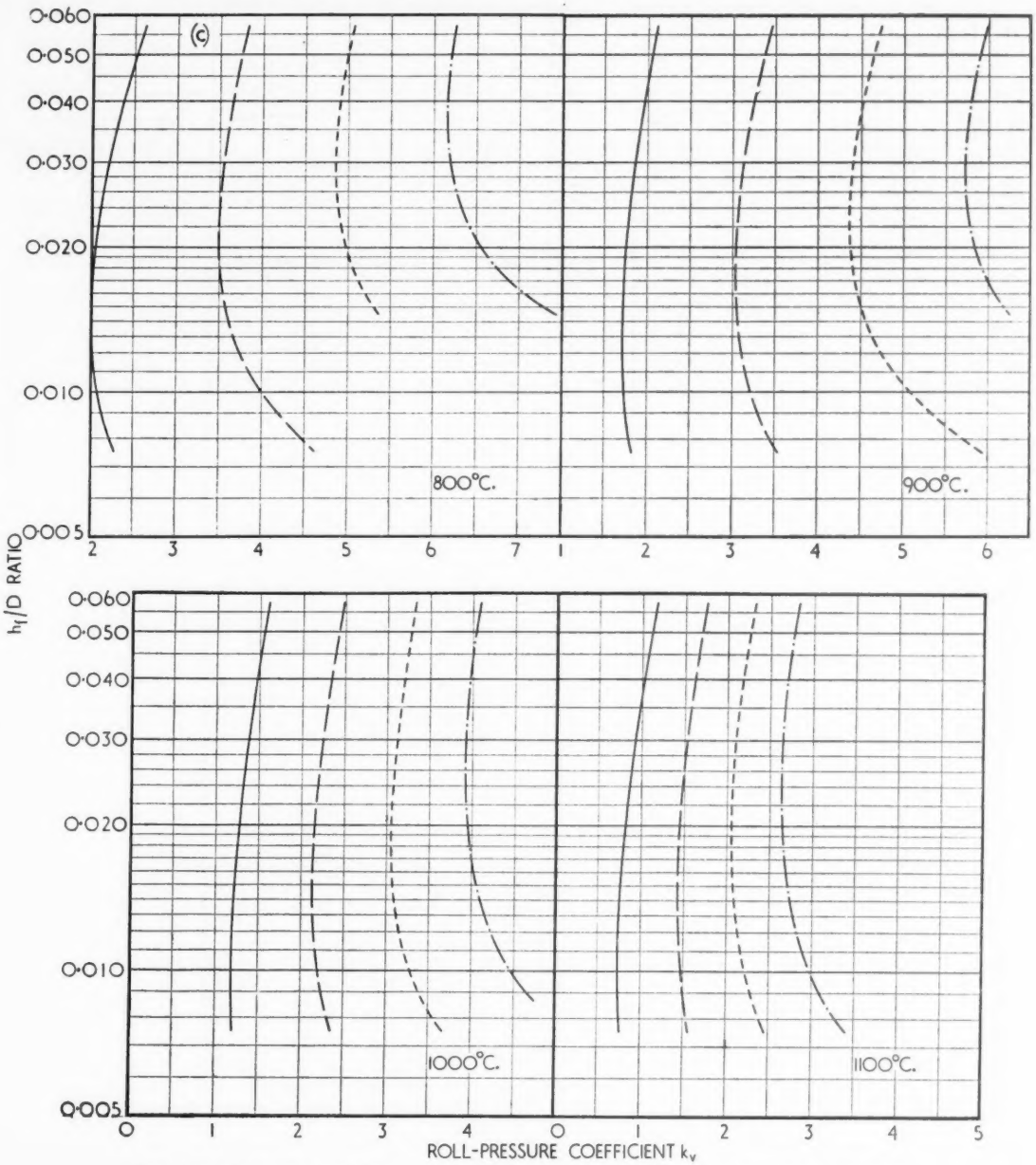
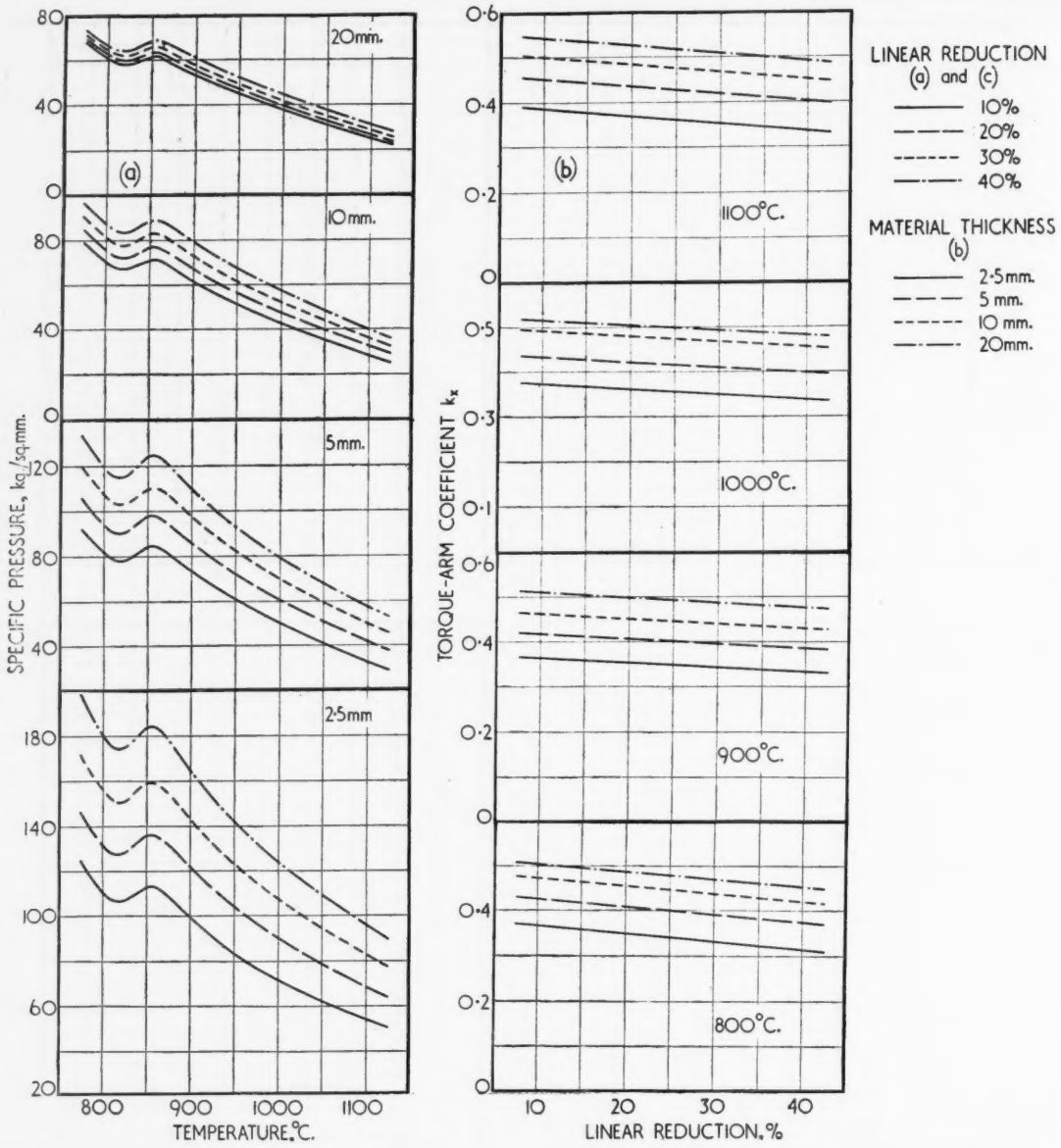


Fig. 12—Effect of steel temperature, reduction, and material thickness for steel 15

great technical difficulties, as was found in the first investigations by Siebel and Lueg,[28] as well as in later work.

Material Thickness—Siebel[25] also studied the influence of material thickness on roll pressure. Generally speaking, the specific roll pressure increases with a decreasing h_f/D ratio, *i.e.*, with lowered material thickness at constant roll diameter. As found for the reduction, the size of the variation is influenced by other factors—steel composition, steel temperature, and reduction. The results obtained by the present author will be evident from the diagrams. For the soft carbon steel 1 and an h_f/D ratio of 0·0074, compared with a ratio of 0·059, the roll pressure is, according to the reduction, 2·3–2·9 times as great at 800° C. and 1·7–3·2 times as great at 1100° C. For the hard carbon steel the corresponding data are 2·2–3·3 and 3·3–4·1. The alloy steels show still higher roll-pressure ratios.

The comments in the previous sub-section about reduction of the material also apply to the material thickness, but the importance of this factor is better



understood as a result of experience gained in rolling strip and sheet.

Roll-Pressure Coefficient

The roll-pressure coefficient, introduced instead of the specific roll pressure, is used to facilitate the calculation of the specific roll pressure, and also to present the resistance to deformation graphically in logarithmic diagrams, which, to a certain extent, eliminates the influence of thickness, bringing out in a better way the two other factors of steel temperature and reduction.

The k_v diagrams of the steels investigated show that the roll pressure calculated on the area $b_m \times D$ instead of $b_m \times x_0$ is in most cases little influenced within a range of h_f/D of 0·01–0·05, *i.e.*, for thicknesses of 15–3 mm. at 300 mm. roll dia. or 40–8 mm. at 800 mm. roll dia. At still lower h_f/D ratios, however, the roll-pressure coefficient starts to rise. The appearance of the diagrams at low thicknesses has not been studied in the present work. An extrapolation of the curves, however, should give valuable information to the rolling-mill engineer interested in thin sections.

Torque-Arm Coefficient

The part of the total rolling torque that consists of deformation resistance and external friction is generally calculated using equation (7), in which $0{\cdot}5x_0$ is chosen as the numerical value of the torque arm x. This implies that the resultant of all the local specific

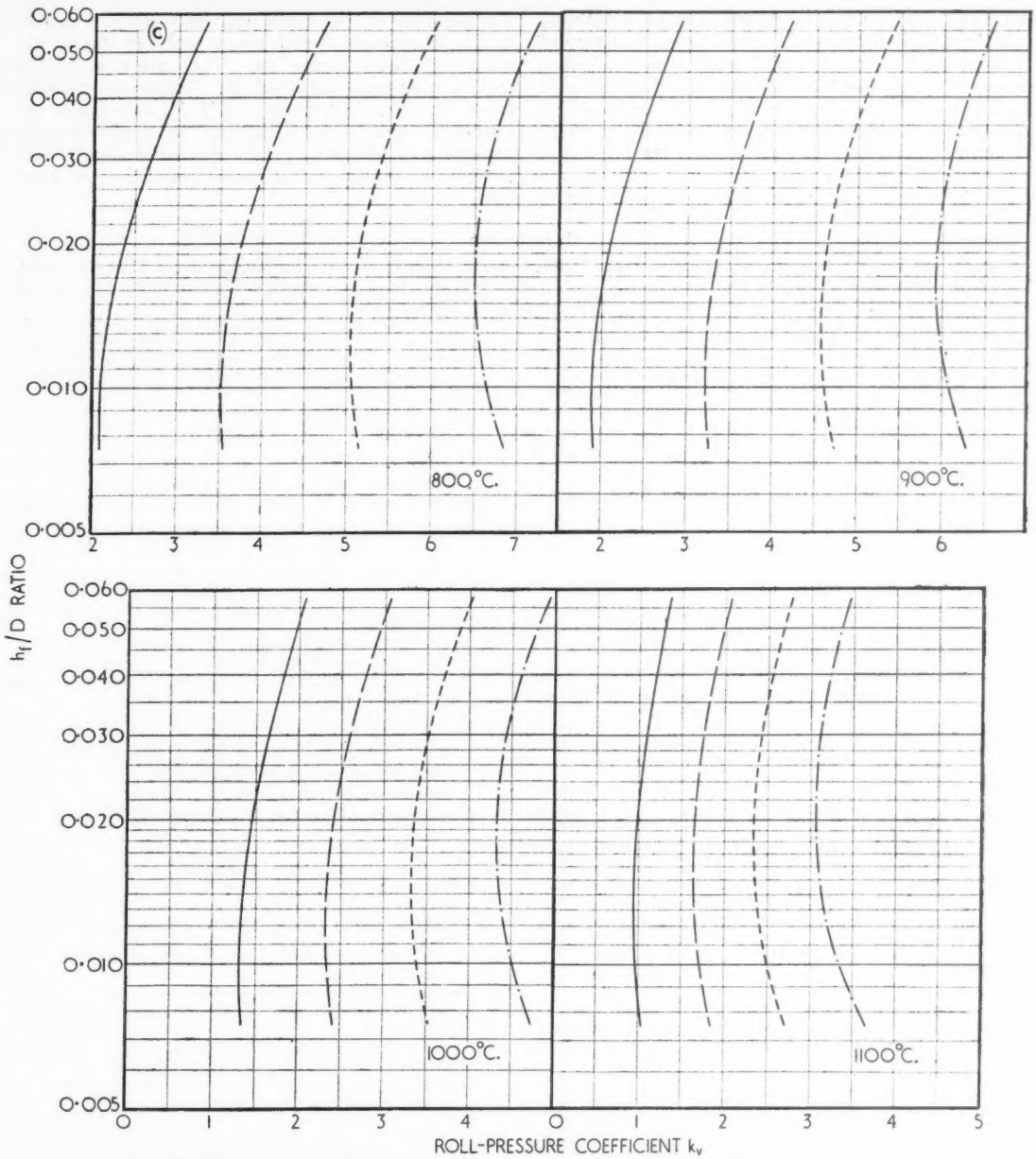


Fig. 13—Effect of steel temperature, reduction, and material thickness for steel 16

roll pressures should always intersect the centre of the projection x_0 of the arc of contact. However, everyone familiar with the hot-rolling process will know that this is not the case. A large part of the present investigation has therefore been devoted to the relationship between torque arm and projection of arc of contact, *i.e.*, with the numerical value of the torque-arm coefficient.

The results obtained, some of which are found in Figs. 9*b*, 10*b*, 11*b*, and 12*b*, show that the value of the coefficient decreases with falling temperature, increasing reduction, and diminishing material thickness. For the soft carbon steel, values are found between about 0·46 and 0·29, and for the hard carbon steel the extreme values are about 0·49 and 0·30. For the alloy steels values from 0·56 to 0·28 have been obtained, which implies that the calculated value for the sum of the torques M_{vd} and M_{vf} in equation (6) have been up to 78% too high when calculated by methods previously used.

Acknowledgments

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Practical Aspects of Hardness Testing of Steel Strip

By G. Molinder

SYNOPSIS

Indentation hardness testing on the surface of steel strip is generally more or less inaccurate because of insufficient thickness of the strip and decarburization or other structural changes in the surface layer.

To determine the real hardness of steel strip a cross-section of the strip must be prepared and the indentation made on that section. The only method that is applicable to such testing is the Vickers, or similar methods, using fairly low loads and having a high precision of aim.

Tensile testing, an indirect method for obtaining accurate hardness values, is limited to material of a hardness lower than about 650 D.P.N.

The Rockwell methods of testing hardened steel strip, not as accurate as the others, can generally only be used on the flat surface of the strip and not on a cross-section. 881

Introduction

No comprehensive study of the specialized subject of the hardness testing of steel strip has, to the author's knowledge, as yet been published. There is a serious lack of precision and uniformity in the two following matters.

Choice of Testing Method—The influence of the thickness of the tested material on the hardness figure obtained is not generally known, and the considerable effect of surface decarburization is not generally taken into account. Cold-rolled steel strip is usually flat and often has a finely polished or ground surface ; there is therefore a tendency to make the hardness indentation directly on the surface of the strip with no specimen preparation. This practice does not always make for accuracy.

Conversion of Hardness Values from One Hardness Scale to Another—Conversion tables published in handbooks and by makers of testing machines do not sufficiently stress that the values they give are only reliable under special circumstances. They may be, say, determined on the basis of measurements on flat and fairly thick specimens, free from decarburization, and are therefore valid only for such specimens.

This paper will discuss the applicability of some generally accepted methods for hardness testing of one category of material, *viz.*, hardened cold-rolled steel strip.

HARDNESS TESTING ON CROSS-SECTION

To ensure correct measurement of the hardness of strip material and elimination of the effect of surface decarburization, a cross-section of the material must first be prepared and then the hardness test must be carried out on that section. The only method then applicable is the Vickers (using ' square ' diamond or Knoop diamond) ; this alone has a sufficiently high precision of aim and is flexible enough to be used for any thickness and hardness. The Vickers hardness value is, within a broad range of loads, independent of the load used.

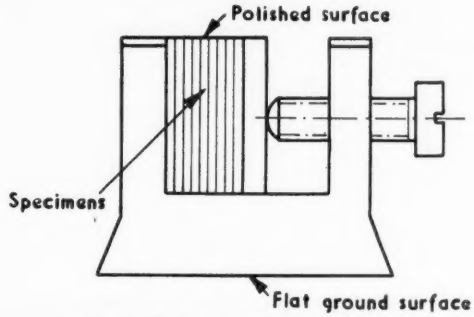

Fig. 1—Fixture for Vickers testing

During recent years the following method of hardness testing has been used by the Uddeholm Co., Munkfors Bruk. Strip specimens with spacers of hardened razor-blade steel are placed in a fixture shown in Fig. 1. The upper surface is ground to the requisite fineness by a flat-grinding machine under a stream of water.

The hardness measurements are made with an original Vickers machine or with a ' 323 ' machine made by Zwick and Co. KG., Einsingen bei Ulm-Donau.

For measuring the impressions or indentations, a higher magnification than that generally obtained in an original Vickers machine is used and a reading of about 500 divisions of the Vickers Armstrong ocular micrometer is aimed at.

The optical system has been calibrated with a special grating made by Siegbahninstitutet, Stockholm. This grating has 144 scratches per mm. and a stated accuracy of 0·15%. There is a good agreement between the original Vickers machine and the Zwick ' 323 ' machine.

When choosing a suitable impression load, two main points must be considered :

(i) The impression must not be too large compared with the strip thickness. Such an impression is obtained when too high a load for the thickness is used, so that the material on both sides of the impression does not give enough support

(ii) The impression must not be so small that the elastic recovery at the corners of the impression becomes too great in relation to the impression-diagonal.

The requisite thickness of the specimen for a reliable hardness measurement on a section has been

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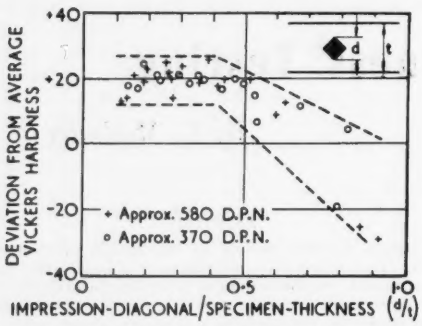

Fig. 2—Deviation from average Vickers hardness *vs.* impression-diagonal/specimen-thickness ratio

determined in the following way. Specimens of a hardened and tempered plain carbon steel with thicknesses of 0·15–0·50 mm. of two different hardnesses (about 580 and 370 D.P.N., respectively) were tested for Vickers hardness as described. Loads of 1–10 kg. were used.

The Vickers (D.P.N.) values thus determined were plotted against d/t where d is the impression-diagonal and t is the specimen thickness.

From Fig. 2 it may be concluded that a value of $d/t = 0{\cdot}4$ is low enough for a reliable Vickers measurement on a cross-section.

The minimum impression-diagonal for which the elastic recovery can be neglected has been calculated from data compiled by Bergsman,[1] and Brown and Ineson,[2] to be 35μ. This implies that strip thinner

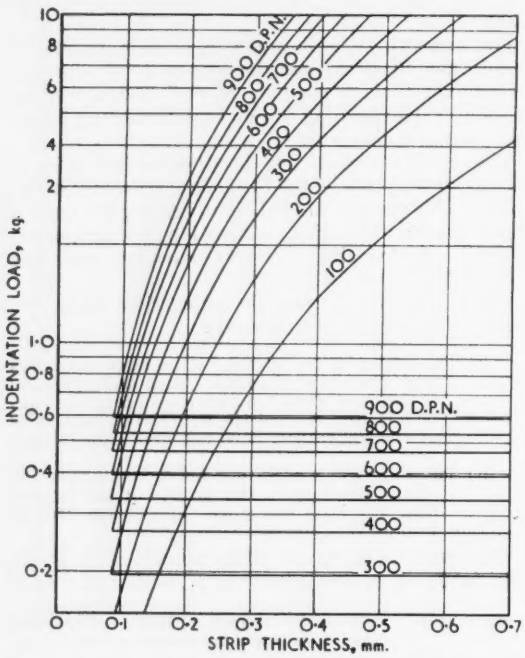

Fig. 3—Maximum and minimum loads for Vickers testing on cross-section

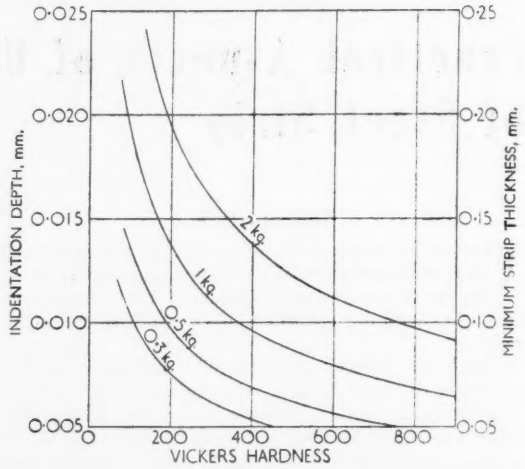

Fig. 4—Minimum dimensions for Vickers testing on strip surface with different loads

than 0·10 mm. cannot be tested on the cross-section, and 0·15 mm. is recommended as a lower thickness limit for such testing. Thinner material may be tested by the Vickers method on the surface after a slight grinding with emery paper or polishing on cloth with aluminium oxide.

Figure 3 gives maximum and minimum loads for Vickers testing on cross-sections at different strip thicknesses, and Fig. 4 shows the maximum load for testing on the surface of the strip.

In the remainder of this paper, Vickers values obtained from testing on cross-section are used as standard when discussing other hardness methods.

HARDNESS TESTING ON SURFACE

Effect of Surface Decarburization

Unless the surface has been very effectively ground or special precautions have been taken during the annealing to restore the carbon content of the surface layer to the average of the strip, hardened steel strip

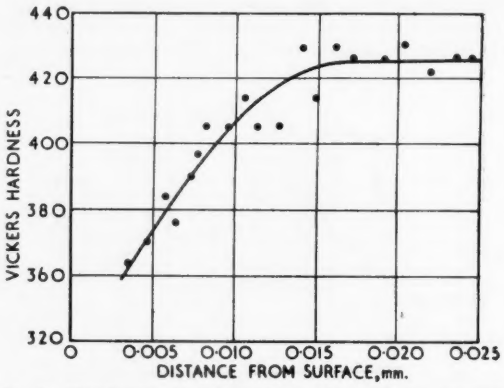

Fig. 5—Hardness variation from the surface inwards of a hardened and tempered 0·50-mm. strip of 0·7% carbon steel

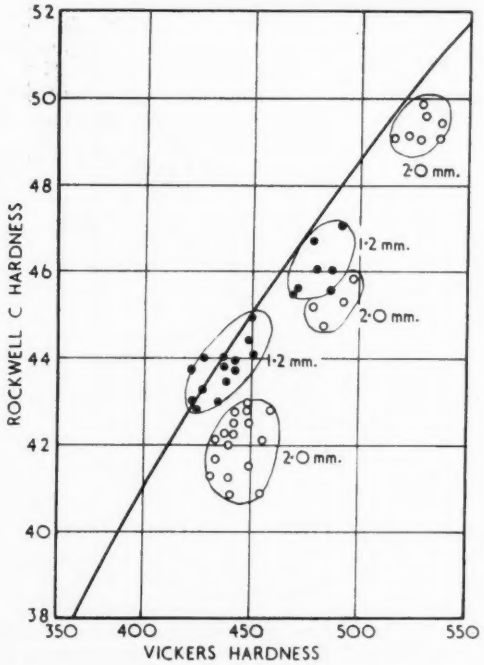

Fig. 6—Rockwell *C* vs. Vickers. Results from testing hardened strip with thicknesses > 1·0 mm. Conversion curve from Rinman and Löwgren[3]

always has a decarburized surface layer of a depth depending on the steel composition and the strip thickness.

Figure 5 shows the variation in hardness from the surface inwards of a hardened and annealed steel strip with a hardness of about 425 D.P.N. measured on cross-section. The material is a 0·50-mm. thick strip of 0·7% carbon steel.

Approximate indentation depths obtained with different hardness testing methods at a hardness of 425 D.P.N. are :

Testing Method	Hardness	Indentation Depth, mm.'
Vickers 50 kg.	425	0·067
,, 10 kg.	425	0·030
,, 2 kg.	425	0·013
Rockwell *C*	41	0·12
,, N_{45}	47	0·053
,, N_{30}	62	0·038
,, N_{15}	82	0·018

Systematic investigations of the effect of decarburization have not been made, but it can be seen from Fig. 5 that such an effect can be appreciable on a hardness value, at least with fairly low loads.

Vickers testing on the surface of steel strip without grinding nearly always gives lower values than those obtained by testing on cross-sections.

Rockwell testing gives generally lower figures than would be expected from the real Vickers hardness of the specimen.

The decarburized layer is generally thicker for thicker material. The influence of decarburization on surface testing therefore becomes greater with increased strip thickness.

Figure 6 illustrates the effect of decarburization when testing fairly thick material. The number of tests is not great but it is shown that Rockwell *C* testing of hardened thick material can give considerable errors, although the material is thick enough for reliable testing.

The inaccuracy due to decarburization is greater when testing methods with lower indentation loads than that for Rockwell *C* are used.

Effect of Strip Thickness

It is also important that excessive loads are not used when hardness testing on the strip surface. Figure 7*a* shows the results of an investigation of the effect of specimen thickness on the Rockwell *C* value, at three different hardnesses. Three series of hardened and ground specimens of a 1·25% carbon steel with an original thickness of 3 mm. were tested. After Vickers and Rockwell *C* testing these specimens were ground to different thicknesses from 3 mm. to 0·4 mm. in a water-cooled flat-grinding machine, and were carefully tested by the Vickers method to ensure that no changes in hardness occurred through heat evolution. After grinding down to the different thicknesses Rockwell *C* hardness tests were carried out.

Figure 7*a* shows that the hardness of the anvil has a strong effect. As might have been expected, a departure from 'real' Rockwell *C* hardness occurs at greater thicknesses for a softer material than for a harder. The minimum thicknesses for reliable Rockwell

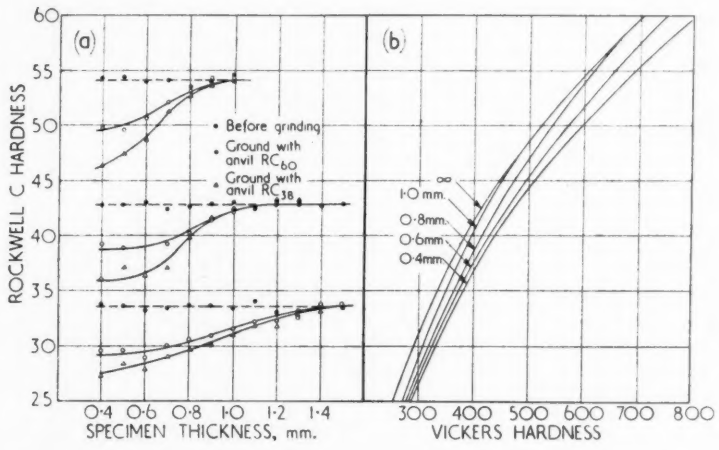

Fig. 7—(*a*) Rockwell *C* vs. specimen thickness at constant Vickers hardness; (*b*) Vickers–Rockwell *C* conversion curves for different thicknesses of specimens

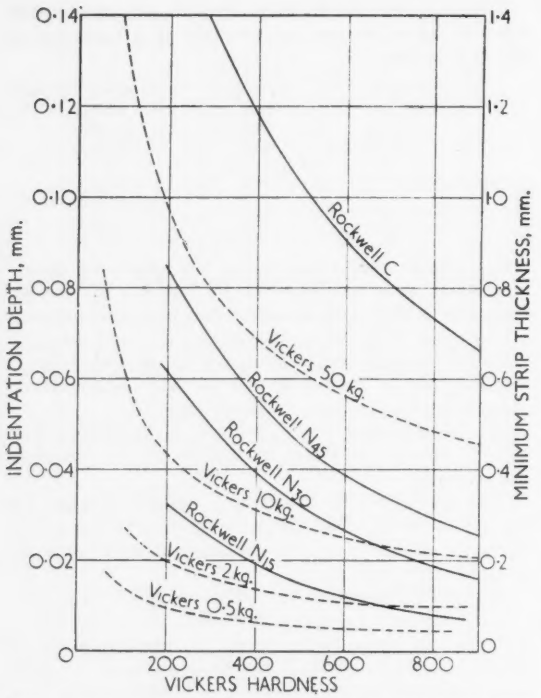

Fig. 8—Indentation depth and requisite thickness for different hardness testing methods

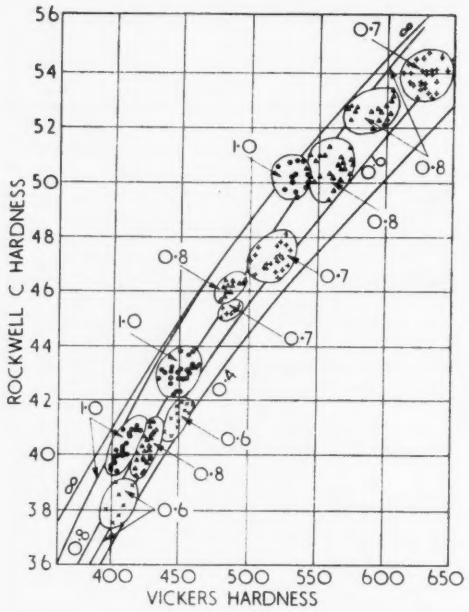

Fig. 9—Rockwell *C vs.* Vickers. Results from testing hardened strip with thickness <1·0 mm.

C hardness tests for the three hardnesses used are :

Hardness, D.P.N. (average)	Rockwell *C* Hardness (average)	Indentation Depth for Rockwell *C*, mm.	Minimum Thickness for Reliable Rockwell *C* Testing, mm.
576	54·0	0·092	0·9
424	42·7	0·115	1·1
307	33·5	0·123	1·3

Thus the thickness of the strip must be about 10 times greater than the indentation depth if a correct figure, convertible to other hardness scales, is to be obtained. If this requirement, which agrees with information given in the "Metals Handbook,"[4] applies to all indentation methods, Fig. 8 can be constructed.

THE ROCKWELL METHODS

Of the hardness scales which are founded on the Rockwell principle, Rockwell *C*, N_{45}, N_{30}, and N_{15} are the most used. Rockwell *BC* (10-kg. preload and 100-kg. total load) and Rockwell *A* (10-kg. preload and 60-kg. total load) are also used to some extent but will not be discussed here.

When hardness testing by Rockwell methods the indentation can generally only be made against the strip surface and not against a cross-section. The dependency of the hardness value on the thickness will be illustrated by some examples from delivery testing by the Rockwell methods.

Figure 9, in which some results of Rockwell *C* testing of hardened steel strip with a maximum thickness of 1·0 mm. are plotted against Vickers values, shows that the Rockwell *C* figures agree fairly well

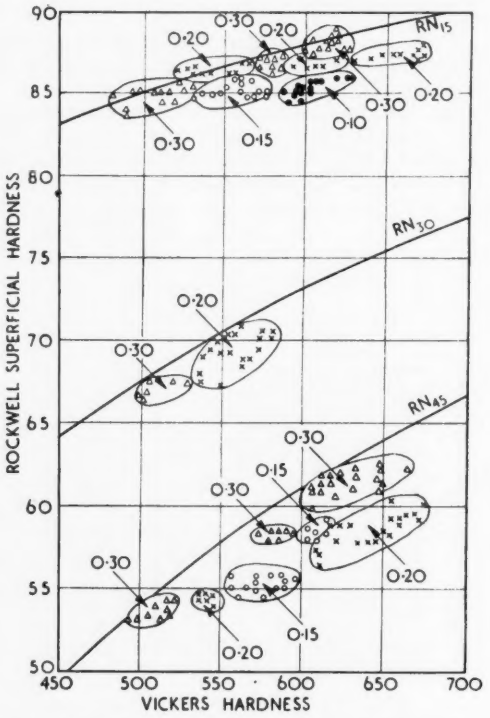

Fig. 10—Rockwell Superficial *vs.* Vickers. Conversion curves from "Metals Handbook"[4]

Table I

ACCURACY OF CONVERSIONS FROM DIFFERENT HARDNESS SCALES TO VICKERS

Conversion	Approx. Accuracy	10 D.P.N. corresponds to:
(1) Tensile strength–Vickers	± 10 D.P.N.	3 kg./sq. mm. at 300 D.P.N. 4 kg./sq. mm. „ 600 „
(2) Rockwell C–Vickers (Fig. 7*b*)	± 20 D.P.N.	1·3 RC units „ 300 D.P.N. 0·6 RC „ „ 600 „
(3) Rockwell C–Vickers (Dependence on thickness not taken into account)*	± 30 D.P.N.	As (2)
(4) Rockwell N_{45}–Vickers	+ 0 — 80 D.P.N.	0·6 RN_{45} at 600 D.P.N.
(5) Rockwell N_{15}–Vickers	+ 0 — 100 D.P.N.	0·3 RN_{15} „ 600 „

* In this case the best conversion is made by a curve lying about 2 Rockwell *C* units below the one published[2, 4]

with the Vickers–Rockwell *C* conversion curves determined for different thicknesses (*see* Fig. 7*b*). No marked tendency is observed for the points to fall below the curves because of decarburization. The decarburized layer seems to be too thin to affect the results significantly. The spread of the points of about ± 1 Rockwell *C* units might, however, be due to the decarburization.

Figure 10 shows the results for corresponding comparisons between Rockwell N_{45}, N_{30}, N_{15}, and Vickers. The Vickers–Rockwell conversion curves have been drawn from data in the "Metals Handbook,"[4] and the machines used give figures in good agreement with these curves when testing thick test blocks.

The departure from the conversion curves is in most cases large. Apart from the fact that for Rockwell N_{45} the thickness of the specimen tested is in many cases less than the requisite thickness according to Fig. 8, the spread in a single test series is large. Thus, for one of the 0·30-mm. groups the same Rockwell N_{45} value (about 61) is obtained although the real Vickers hardness varies from 600 to 660 D.P.N.

For Rockwell N_{15} the spread is great, although all thicknesses except the 0·10-mm. group are sufficient for reliable testing. For the three groups of 0·20-mm. specimens a value of about 86 Rockwell N_{15} has been obtained irrespective of the fact that the Vickers hardness of the strip section varies from about 520 to about 650 D.P.N. The value of such testing is questionable.

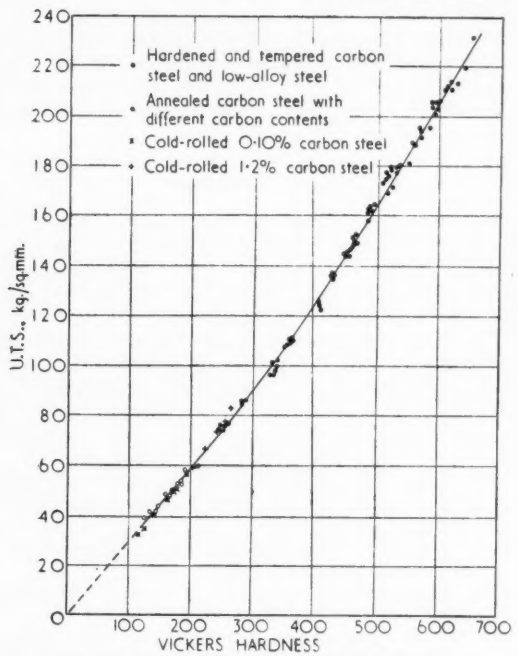

Fig. 11—U.T.S. *vs.* Vickers hardness

TENSILE TESTING

In most cases an accurate determination of the real hardness of a material can be made by a tensile test, at least when the hardness is lower than about 650 D.P.N. (about 260 kg./sq. mm. U.T.S.). The U.T.S. is practically unaffected by a decarburization of normal depth.

In Fig. 11 corresponding values obtained from tensile strength and Vickers (on cross-sections) are plotted for plain carbon steels and low-alloy steels with <2% Si, <0·9% Mn, and <2 %Ni. The points fit very well to a curve. Maximum departure from this curve is about ±10 D.P.N. The spread tends to become greater with increasing hardness.[6]

CONVERSION OF HARDNESS FIGURES

Published conversion tables* (which sometimes differ from each other) for different hardness scales have presumably been compiled from measurements made on flat and sufficiently thick test blocks of even hardness and fine surface. Thus, they are only to a limited extent applicable to a conversion of hardness values obtained from strip steel.

A conversion to Vickers of, *e.g.*, a Rockwell N_{45} value determined on a hardened strip can give large errors. A conversion of the same figure to Rockwell *C* might be still more inaccurate.

Because the limitations of the different methods are not generally known or taken into account complete confidence often exists in the conversion tables; much misunderstanding is thereby caused between makers and users of cold-rolled steel strip.

Some approximate figures for the accuracy of conversions from different hardness scales to Vickers are given in Table I.

*In handbooks such as the "Metals Handbook", "S.A.E. Handbook", British Standards, "Werkstoffhandbuch Stahl und Eisen", and published by makers of hardness-testing machines.

Table II

RELIABILITY GRADING OF CONVERSIONS

A = Accurate conversion
B = Fairly accurate conversions; can be used if errors of about ±20 D.P.N. are tolerated
C = Inaccurate conversions; errors of about ±40 D.P.N.
X = Completely worthless conversions.

From \ To	Vickers (on cross-section)	U.T.S.	Rockwell *C*	Rockwell N_{45}	Rockwell N_{30}	Rockwell N_{15}
Vickers (on cross-section)	—	A	B	C	C	X
U.T.S.	A	—	B	C	C	X
Rockwell *C*	B	B	—	X	X	X
Rockwell N_{45}	C	C	X	—	X	X
Rockwell N_{30}	C	C	X	X	—	X
Rockwell N_{15}	X	X	X	X	X	—

These figures show plainly the risks arising from a conversion of a Rockwell figure. An uncertainty of 50–100 D.P.N. can rarely be tolerated. The uncertainty might be even greater in converting from one Rockwell scale to another.

In Table II an attempt has been made to introduce a grading of the different conversions.

RECOMMENDED METHODS FOR HARDNESS TESTING

For correctness and accuracy the Vickers method (or similar methods) on a cross-section must be used. The apparatus and recommendations for the choice of suitable load have been discussed. Strip thinner than 0·15 mm., however, should not be tested on a cross-section, but on the strip surface after light grinding with emery paper or polishing. The indentation load must be chosen so that the impression will be neither too small (*i.e.*, the diagonal should be $>35\mu$) nor too large.

An accurate Vickers test requires fairly high precision of method and equipment. When using loads lower than about 10 kg. a larger magnification than that used in the original Vickers machine is recommended.

A person trained in Vickers testing and using perfect tools, however, can carry out a determination of Vickers hardness, including putting the specimen into a fixture, indentation, and measurement, very rapidly (5 min. per specimen with 3 impressions on each). Another advantage of the Vickers method is that a single scale covers all hardnesses encountered in steel treatment.

An accurate indirect method of hardness testing is the determination of U.T.S. A tensile test can easily be made when the material is fairly thin and so narrow that no slitting is required. With thickness below about 2 mm. and hardness below about 500 D.P.N. (*i.e.*, about 170 kg./sq. mm.) this test is simple and accurate. With greater thickness the specimen preparation might be somewhat laborious. At hardnesses above 500 D.P.N. great care must be taken to polish the edges of the specimen and centre the specimen in the tensile testing machine.

Rockwell *C* testing against the surface of the strip can be made with fairly good accuracy when the specimen thickness is greater than about 0·5 mm. (Placing two thin strips together, a method sometimes used, is objectionable.) The variation with increased thickness must then be taken into consideration. Material thicker than about 1·0 mm. should be ground on the surface before testing, either with emery paper or with a grinding wheel and careful water-cooling ; grinding must be deep enough for all decarburization to be removed but not so deep that the material becomes too thin for a reliable measurement. The Rockwell *C* method is simple and rapid.

The Rockwell N_{45}, N_{30}, and N_{15} methods give fairly inaccurate results ; they can theoretically be used for strip thinner than 0·50 mm. but cannot be recommended. The surface can be ground before testing to eliminate the decarburization layer but this is not advisable as the testing methods are affected by thickness. In addition, the Rockwell Superficial methods give poor differentiation ; 100 D.P.N. correspond to only about 3 Rockwell N_{15} units.

In some cases a Rockwell Superficial value, however, gives an acceptable description of a material, *e.g.*, for a strip product of uniform dimensions and surface finish. The original Rockwell value should then be quoted without any conversion to other scales.

Reliable methods other than Vickers (on cross-sections), tensile testing, and to some extent Rockwell *C*, do not exist ; the other Rockwell methods or such methods as scleroscope testing are generally not accurate enough.

Acknowledgments

The investigation has been made in the Munkfors Steelworks Laboratory of the Uddeholm Company, Sweden. Further work on the problem of hardness testing of steel strip is carried on in the research programme of Jernkontoret.

The author wishes to express his thanks to the Uddeholm Co. for permission to publish this paper and to Mr. Erik Nygren, Munkfors, for valuable contributions and advice.

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Formation of Martensite in Austenitic Stainless Steels

EFFECTS OF DEFORMATION, TEMPERATURE, AND COMPOSITION

By Tryggve Angel

SYNOPSIS

The isothermal formation of martensite induced by plastic deformation was studied in austenitic stainless steels in the temperature range —188 to 100° C. as a function of stress, strain, and deformation energy. The influence of temperature is discussed in terms of the relation between the martensitic and the slip mode of deformation. The main transition between the two modes was found to take place within a narrow temperature range. In this range a modified version of M_d, termed M_{d30}, was chosen, giving a measure of the austenite stability easier to determine practically. The effect of composition on the austenite stability in regard to martensite formation was evaluated statistically. It was found that the action of Cr, Si, and Mo was the same as that of Ni, Mn, C, and N_2, in that they all tend to stabilize the austenite. Finally, the anomalous increase in stress observed in the stress/strain curve was found to be proportional to the amount of martensite formed and also to the carbon content of the martensite. 882

Introduction

As is well known, the transformation of austenite to martensite is the basic reaction in the hardening of carbon steels. It is less well known, however, that this transformation may also play an important rôle in the mechanical working of austenitic steels, and is often encountered in practice as an unusual work hardening. The austenitic stainless steels of the 18/8 chromium–nickel type are of particular importance, and the formation of martensite in these steels is the subject of the investigation described in this paper. The general characteristics of the martensitic transformation are:

(i) It is diffusionless, *i.e.*, the transformation product has identically the same composition as the matrix (austenite in the case of steel)
(ii) It occurs primarily on cooling
(iii) The temperature, M_s, at which it starts is practically independent of the cooling rate
(iv) A definite crystallographic orientation relationship exists between the lattice of the martensite and that of the matrix
(v) Transformation can be made to occur at temperatures above M_s [1–9] under the influence of mechanical stresses and strains.

According to Cohen *et al.* the martensitic transformation is a nucleation-and-shear process with strain embryos as starting points for the transformation. This can be described as a shear transformation characterized by a homogeneous shear of the austenite lattice along an invariant plane (the habit plane) and a dilatation.[10] Transformation can be regarded as taking place almost instantaneously, as the formation of a martensite crystal occurs in about 3×10^{-7} sec. [11]

For the martensitic transformation, as for any other reaction, the following two conditions must be fulfilled:

(i) The free energy of the system must decrease during the transformation
(ii) Nuclei must be present.

Cohen *et al.*[12] suggested that screw dislocations constitute nuclei for the martensite formation. This was later proved by Krisement[13] in a theoretical study on martensite with a (225) habit. As a result of the large number of dislocations that exist even in a well-annealed austenitic structure, more embryos are generally present than are necessary for the reaction to occur. The controlling factor for the kinetics of the transformation, therefore, is not the rate of nulceation but the supply of free energy.[3]

The free-energy change of the system must be large enough to enable the reaction to mount the activation barrier between the austenitic and the martensitic states and also to supply energy to be bound as surface energy, elastic and kinetic energy, etc. Because of these and other hindrances to the reaction, martensite does not form spontaneously at the thermodynamic equilibrium temperature T_E, but only after a certain amount of undercooling, usually about 200–300° C. and corresponding to a free-energy change of about 200–400 cal./mol., has taken place.[4, 12]

As has been stated, martensite formation can be made to occur at temperatures above M_s by deformation of the austenite.[1, 2] Scheil investigated an iron–nickel alloy containing 29% of nickel, and found that the amount of martensite increased with the degree of cold working and decreased with increasing working temperature.

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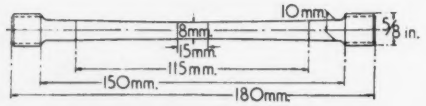

Fig. 1—Tapered tensile test bar

Above a certain temperature M_d no amount of deformation can bring about transformation. It has been suggested[12] that M_d is identical with the equilibrium temperature T_E mentioned above.

As the martensitic transformation is associated with a shear strain (of about 0·20) it may be regarded as a deformation process that competes with the usual slip process.[1, 7] With decreasing temperature the critical resolved shear stress for slip, *i.e.*, the resistance to slip, increases, whereas the resistance to martensite formation decreases. Above a certain temperature the resistance to slip is less than the resistance to martensite formation and slip is therefore the dominating process of deformation. Scheil suggested a critical resolved shear stress along a potential habit plane as a criterion for the formation of martensite induced by deformation. He also pointed out that not only the shear stresses but also the normal stresses might be important. Cohen *et al.*[7] showed experimentally the influence of the normal stresses. A thermodynamical criterion has recently been formulated[4] for the formation of martensite, taking into consideration the contribution of mechanical energy which deformation makes to the chemical free energy of the reaction. The transformation is aided by shear stresses but may be aided or opposed by the normal stress component, depending on whether the latter is tensile or compressive. This criterion successfully explains the influence of elastic stresses on M_s.

Investigations within the plastic range[2, 14-19] have given a qualitative picture of how different factors, such as degree of deformation, working temperature, and chemical composition of the austenite, affect the austenite–martensite transformation. The present investigation was undertaken to obtain more quantitative information about the interrelationship of these factors, especially in regard to the commercially important austenitic stainless steels.

EXPERIMENTAL DETAILS

A number of test bars were deformed isothermally and the amount of martensite formed was determined as a function of strain, stress, and deformation energy. The effect of temperature and of chemical composition was also investigated.

Material and Heat-Treatment

Several heats of the general 18/8-type steels were taken from current production, and 11-in. ingots were rolled down to 1-in. square bars, heat-treated at 1050° C. for ½ hr., and water-quenched. The grain size after heat-treatment was about A.S.T.M. No. 6.

Equipment and Procedure

The specimens used were in the form of tapered tensile test bars[6, 21] (Fig. 1). Two fine reference marks, a spiral scratch with a pitch of 4·00 mm. and a longitudinal scratch, were made on each bar. The diameters of the bar at the intersections of these two scratches, and the lengths l_0 and l, before and after the bar was pulled to fracture, were measured with an accuracy of $\pm$ 0·01 mm. It was thus possible to calculate the maximum true stress and true strain within each section.

The true strain is

$$\epsilon = \ln\frac{l}{l_0} \quad \ldots\ldots(1)$$

or, assuming constant volume,

$$\epsilon = \frac{A_0}{A}$$

where A_0 and A are the cross-sectional areas of the specimen before and after testing. However, the volume cannot be regarded as constant during deformation, as the latter results in a transformation of austenite to martensite which is associated with a dilatation. Let the linear dilatation be α; then the true strain can be written

$$\epsilon = \ln\frac{l}{l_0(1+\alpha)} \quad \text{or} \quad \epsilon = \ln\frac{A_0(1+\alpha)^2}{A}$$

Rearranging:

$$\epsilon = \ln\frac{l}{l_0} - \ln(1-\alpha) \quad \text{or} \quad \epsilon = \ln\frac{A_0}{A} + 2\ln(1+\alpha)$$

Eliminating $\ln(1+\alpha)$:

$$\epsilon = \tfrac{1}{3}\left(\ln\frac{A_0}{A} + 2\ln\frac{l}{l_0}\right) \quad \ldots\ldots(2)$$

This is the expression for the true strain taking into account the increase in total volume due to transformation. The error in using equation (1) instead of equation (2) was found to be about 0·005–0·01 units of strain, which was considered to be negligible in comparison with the large strains measured, which were generally 10–100 times greater. The strain was therefore calculated according to equation (1).

The test bars were pulled in an Alpha 75-ton tensile testing machine at a speed of 1 mm./min. They were usually pulled to fracture, one bar being tested at each temperature. In some cases it was necessary to test a second bar at a lower load, to extend the results obtained with the first bar over a wider strain range. To enable isothermal runs to be carried out at different temperatures and to absorb the heat of deformation evolved, the test bars were surrounded by a Dewar container welded to the lower grip of the machine. The container was filled with liquid air for the experiments at — 188° C. and with dry ice and alcohol for temperatures between -- 70° C. and room temperature. For higher temperatures, oil heated by an immersion heater was used. The liquid was agitated by a propeller and the temperature was kept constant within $\pm$ 0·5° C.

After extension the bars were cut with a thin milling cutter at the intersections of the two scratches into pieces about 4 mm. long. Deformation and martensite content were regarded as constant within these small pieces and equal to the measured average values.

Determination of Martensite Content

The proportion of martensite in each sample was measured at room temperature in a magnetic balance,[20] as the specific saturation magnetization σ_s, at a field

Table I

COMPOSITIONS AND PROPERTIES OF STEELS EXAMINED

Heat No.	Chemical Analysis							M_{d30}		M_S, °C.	σ_0, c.g.s. units
	C, %	N, %	Si, %	Mn, %	Cr, %	Ni, %	Mo, %	Exper., °C.	Calc., °C.		
H4531	0·06	0·024	0·39	0·33	18·4	8·6	0·06	34	33	...	161·3
X7522	0·09	0·053	0·42	0·42	18·2	8·6	0·29	− 1	3	<−183	160·4*
R7923	0·09	0·020	0·43	0·48	18·2	8·7	0·05	30	21	−170†	161·5
MH1155	0·13	0·038	0·37	0·50	17·5	8·0	0·06	− 5	11	<−183	162·8
MH1163	0·12	0·013	0·81	0·95	19·2	8·4	0·07	−15	− 8	<−183	157·7
H4524	0·14	0·022	0·28	0·45	18·2	7·9	0·12	14	5	<−183	162·0
E1473	0·16	0·018	0·55	0·36	18·2	7·6	0·08	0	0	...	159·8
MH1159	0·19	0·017	0·51	0·50	17·7	8·1	0·06	0	−12	<−183	161·6
MH1158	0·14	0·015	0·48	0·45	18·2	8·2	0·75	− 1	− 8	<−183	159·9
MH1162	0·14	0·010	0·50	0·98	18·4	8·0	0·77	−16	−11	<−183	157·2
MH1157	0·16	0·019	0·44	0·48	16·6	7·9	0·73	2	7	<−183	163·2
MH1156	0·24	0·031	0·52	0·44	15·3	8·1	0·77	−12	−21	<−183	164·6
R37683	0·10	0·044	0·42	7·00	15·1	5·2	...	27	30	−175†	
S76219	0·11	0·029	0·19	6·95	15·6	4·2	0·08	38	36	−170†	
HA25531	0·14	0·023	0·33	8·90	15·9	5·1	...	2	− 3	...	
HA25526	0·15	0·018	0·38	9·10	16·1	5·1	...	−17	−10	...	

* Determined experimentally; all other values calculated according to equation (4)
† Approximate

strength of about 12,000 oersted. The accuracy of this method was found after several duplicate runs to be within $\pm 0{\cdot}5$ c.g.s. units. The relative values thus obtained were converted to percentages of martensite according to the following method, developed by Hedebrant.[22]

For one of the heats the integrated X-ray intensity for the (111) line of the austenite and the (110), (101) double line of the martensite was measured in a series of samples with varying martensite content. This again gave relative values for the amount of martensite, which were plotted against the σ_s values previously obtained, to give by extrapolation the value of σ_s for 100% martensite, σ_0. As σ_s is directly proportional to the percentage of martensite M,

$$M = \frac{\sigma_s}{\sigma_0}.100 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3)$$

For the heat investigated, *X*7522 (Table I), σ_0 was found to be $160{\cdot}4 \pm 4$ c.g.s. units. For other heats the influence of compositional changes on σ_0 was determined according to the formula:

$$\sigma_0 = 160{\cdot}4 + \Sigma \Delta N_i \Delta\sigma_{0i} \ldots\ldots\ldots\ldots\ldots\ldots(4)$$

where ΔN_i is the difference in content of alloying elements between heat *X*7522 and the heat considered, and $\Delta\sigma_{0i}$ is the change in σ_0 of iron per 1% addition of different elements.[23] The values thus calculated are given in the last column of Table I.

To check the possible effect on the content of martensite of the cold-working induced by the milling operation, a test bar was deformed and measured in the unsectioned condition, and was remeasured after cutting. No significant differences were observed in the σ_s values obtained before and after cutting and it was concluded that the effect of the milling operation was negligible.

Preparation of Specimens for Electron Microscopy

Cross-sectional samples were taken from deformed test bars, ground, and polished electrolytically. After etching they were repolished several times to ensure that no cold-working effect remained after the grinding operation.

A 5% hydrochloric acid solution in alcohol with 1·5% picric acid was used for electrolytic etching; a current density of about 20 amp./sq. cm. was used, with the specimen as a cathode. The etching was carried out for periods of 1 sec., repeated 10 times.

After etching, replicas were made from a 0·8% solution of Mowital F40 in chloroform. No shadowing was applied. The electron microscope used was an R.C.A. microscope model E.M.U.–2C.

EXPERIMENTAL RESULTS

A large number of curves were obtained for the deformation tests but, as these were very similar in form from one heat to another, they are not all presented in this paper. A limited number of diagrams from a single heat, representing the general type 18/8 Cr–Ni stainless steel, were chosen to show the typical features. The individual characteristics of each heat are given as M_{d30} values (*see* definition below) in Table I and are only illustrated graphically where this is necessary for the discussion.

The Effect of Deformation

A typical set of curves relating martensite content to true strain is shown in Fig. 2. The expression found to fit these curves was

$$\ln \frac{f}{1-f} = n \ln \epsilon + k \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(5)$$

where the fraction transformed $f = M/M_T$, and n and k are constants. Equation (5) is of the log-autocatalytic type given by Austin and Rickett[25] for the isothermal transformation of austenite, with strain substituted for time. When the curves were plotted on a log/log scale, a series of virtually parallel straight lines was obtained. The slope n was approximately equal to 3 and was independent of chemical composition and temperature. The constant k, however, varied with both these factors.

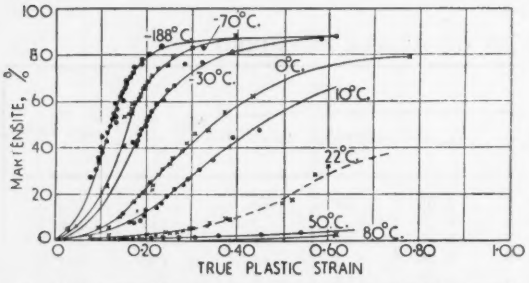

Fig. 2—Formation of martensite by plastic tensile strain at various deformation temperatures

The slope of the curves in Fig. 2, *i.e.*, the rate of reaction $dM/d\varepsilon$, is zero at the beginning and gradually increases to a maximum after a considerable amount of strain. After this the rate gradually decreases until no further transformation occurs upon further straining, and a limiting value for the martensite content, M_T, is reached. This value is lower the higher the temperature, and becomes zero above a certain temperature which according to definition is the M_d temperature. It was also observed that M_T in no case exceeded about 90% of martensite.

The decreasing reaction rate $dM/d\varepsilon$ can be regarded as depending either on a stabilizing effect of deformation on the austenite, or on the decreasing amount of austenite available for transformation. Accordingly, the rate of reaction per unit of austenite,

$$\frac{100}{100 - M} \times \frac{dM}{d\varepsilon}$$

was plotted against the percentage of martensite M (*see* Fig. 3). It is clear that a stabilizing effect is operative during the later stages of transformation and also that there is a stimulating effect in the beginning. The degree of transformation is plotted as a function of true stress in Fig. 4; this shows that transformation does not start until a certain value of the stress has been reached. This threshold value is generally higher the higher the temperature and is also a function of the chemical composition, increasing with increasing austenite stability. An anomaly is shown by the form of the curve for — 188° C., which starts at a higher stress than those for — 70° C. and 0° C. but later intersects these curves. This point will be discussed further below.

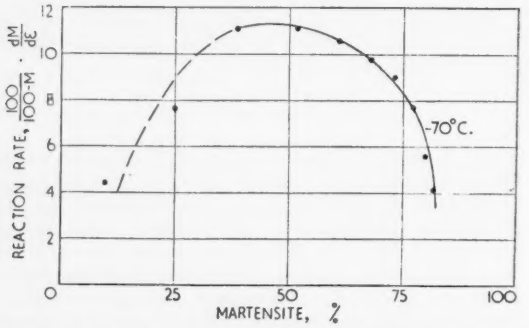

Fig. 3—Rate of reaction per unit of austenite as a function of martensite content

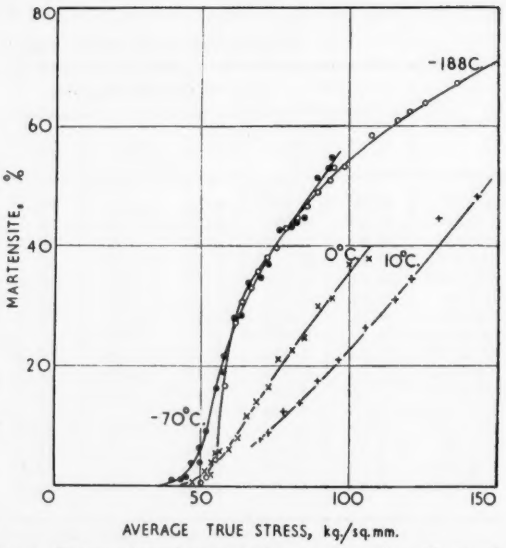

Fig. 4—Formation of martensite as a function of average true stress at various deformation temperatures

A pertinent question is whether the governing factor in deformationally induced transformation is the formation of new nuclei or the action of stress itself. New nuclei may be formed in the straining of the material, as the number of dislocations increases from about 10^8/sq. cm. in a well-annealed structure to about 10^{12}/sq. cm. in a heavily cold-worked material. To investigate this question a number of cylindrical tensile test bars were pulled at room temperature to

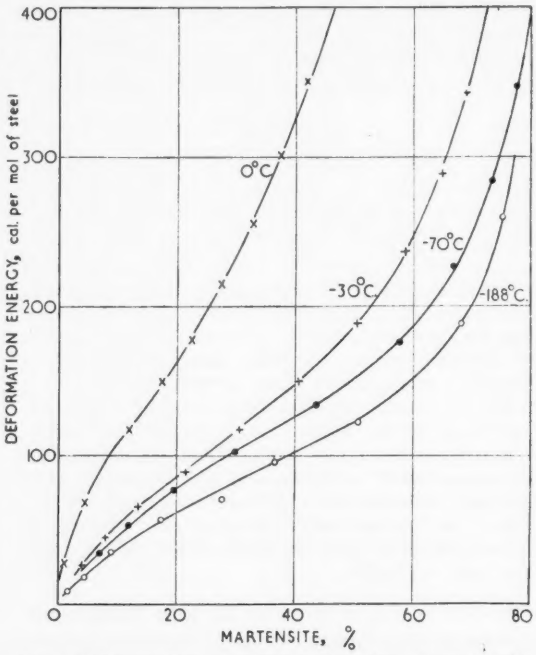

Fig. 5—Deformation energy as a function of martensite content at various deformation temperatures

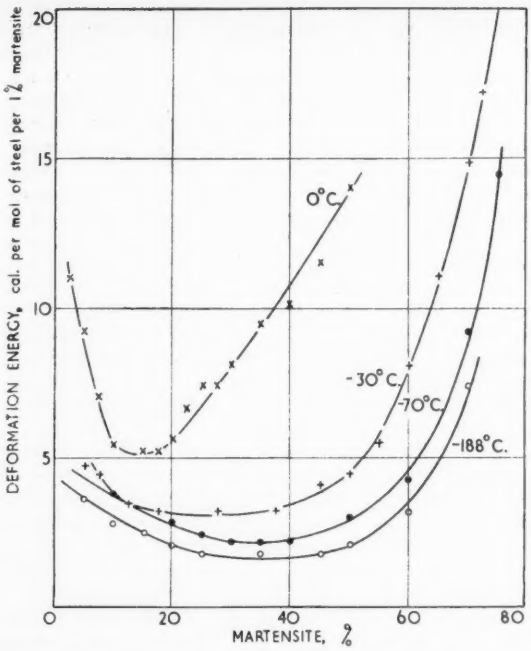

Fig. 6—Deformation energy required to form 1% of martensite at various temperatures

varying strains up to 0·44 and immediately quenched to — 185° C. No transformation took place on cooling and it was therefore concluded that transformation was not due to strain alone.

As has been mentioned, the true-stress/true-strain curve may be evaluated from measurements on the tapered test bars. Integrating the area under this curve and taking the corresponding values of martensite content, the deformation-energy/transformation curves may be obtained (Fig. 5). These curves show a course of transformation resembling that shown by the transformation/strain curves (Fig. 2).

To determine the energy required for the formation of 1% of martensite, the curves in Fig. 5 were differentiated graphically; the results are shown in Fig. 6. A stimulating effect is again observed in the early stages of the transformation. This stimulating effect changes to a retarding one after a degree of transformation that is lower the higher the temperature.

Effect of Temperature

It is clear from Figs. 2–6 that temperature has a strong influence on the course of transformation; Figs. 7 and 8 illustrate this temperature dependance further. Figure 7 shows that there is an abrupt change in stability within a rather limited temperature range. At low strains the curves tend towards a certain temperature above which no transformation appears to occur. McReynolds identified this temperature with M_d;[9] Starr[26] questioned this, as he obtained martensite in cold rolling at temperatures well above this 'plastic critical temperature.' Figure 7 shows that transformation does occur at higher temperatures, provided that the strain is large enough. The

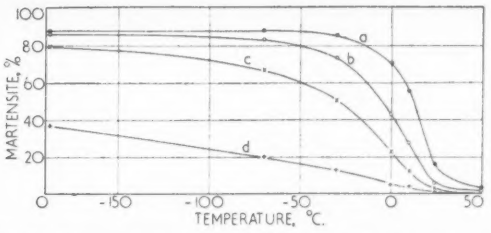

Fig. 7—Temperature dependence of martensite formation at true plastic strains of (*a*) 0·50, (*b*) 0·30, (*c*) 0·20, (*d*) 0·10

concept of a true 'plastic critical temperature' might more suitably be replaced by that of a 'transition temperature,' *i.e.*, the temperature of transition from slip to martensite formation as the primary mechanism of deformation. It has been suggested[27] that M_d is a better measure of the stability of austenitic stainless steels than M_s can be. The M_d temperature as defined is, however, difficult to determine experimentally. In this paper, therefore, a modified definition of M_d has been chosen, coincident with the transition temperature mentioned. According to this definition, which is, of course, completely arbitrary, the characteristic temperature is the temperature at which 50% of martensite is formed in tension after a true strain of 0·30. This temperature is termed M_{d30}. It appears from what is known about the transformation mechanism that the characteristic temperature will vary with the method of deformation, *e.g.*, different values will be obtained for deformation by tension and by compression.

Figure 8 shows that above M_{d30} the energy of deformation, the stress, and the strain, when 10% of martensite has been formed, rapidly increase towards infinity. Values at 10% of martensite were chosen rather than at 0%, as the onset of transformation is difficult to determine accurately.

Electron Microscopy

According to the theory put forward in this paper, it is to be expected that the amount of slip in relation to the amount of martensite formation increases with increasing temperature, and this question was investigated by means of the electron microscope. Replicas

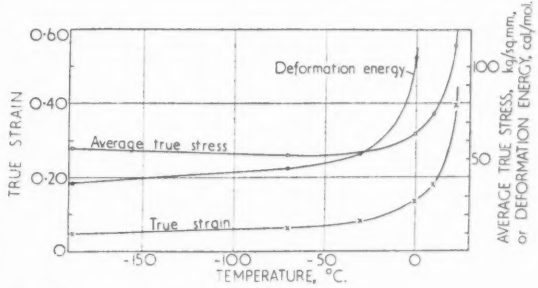

Fig. 8—Deformation energy, average true stress, and true strain required to start transformation (taken at 10% martensite) in tension, as a function of temperature

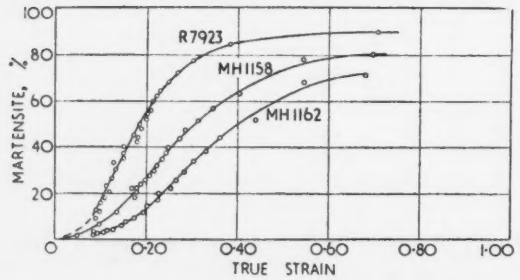

Fig. 9—Transformation curves at 0° C. for three steels of different austenite stability

were made from specimens from a number of test bars deformed at various temperatures; several bars were deformed to the same total strain but to give varying amounts of martensite, and in others equal amounts of martensite were produced by varying degrees of deformation. In the electron micrographs the martensite appears dark against the light austenite. Figure 11 is from a test bar deformed at 10° C. The upper part of the large austenite area in Fig. 11*a* is shown at a higher magnification in Fig. 11*b*. This shows a system of slip lines, which are probably accompanied by some martensite formation (the dark-etching spots). After the first slip system was operative, slip occurred along another slip system, which gave the first-formed slip lines their bent shape.

In a similar study of a test bar deformed at — 188° C. no slip or only slight traces could be detected. Figures 12*a* and *b* are from a test bar deformed to a true strain of 0·14 at this temperature. A preferred orientation of the martensite is clearly visible.

It is interesting to compare Figs. 11, 13, and 14, from specimens which were deformed to approximately the same extent at 10°, — 30°, and — 188° C. The martensite content increases from 16% at 10° C. to 83% at — 188° C. At 10° C. traces of slip were clearly visible, but with decreasing temperature they became fewer. At — 188° C. practically no slip was observed at a strain of 0·14. At a strain of 0·23 the structure has changed to a rather undefined appearance (Fig. 14) in contrast to the clear structure obtained at lower strains (Fig. 12). As the martensite content is 83% and the rate of reaction is very low at this stage, it is assumed that the resistance to deformation of the austenite has become equal to that of the martensite. It therefore appears that at the final stage of transformation three fundamental processes of deformation are operative: martensite formation, slip in the austenite, and slip in the martensite.

The electron-microscope study has thus largely confirmed the theory of a transition from deformation by slip to that by martensite formation with decreasing temperature.

Effect of Composition

Figure 9 shows qualitatively the effect of chemical composition on the stability of the austenite. A more quantitative treatment is possible with Fig. 10, from which the M_{d30} values can be obtained. Before this is discussed, however, some remarks about the term 'stability' may be appropriate. This term, as has recently been pointed out,[27] has been applied rather loosely to two different concepts. In one sense it has been used to denote the stability of austenite in regard to the formation of δ-ferrite at high temperatures. In this connection silicon, chromium, molybdenum, etc., are said to be ferrite-forming elements, whereas carbon, manganese, nickel, and nitrogen are austenite-stabilizing elements. In another sense, the term is used, as in this paper, for the stability of austenite in regard to the formation of martensite at considerably lower temperatures, either spontaneously or by deformation. In this reaction, the behaviour of the elements is quite different and, with some possible exceptions, they all tend to stabilize the austenite; a strong δ-ferrite-forming element such as chromium, for instance, acts as a strong austenite-stabilizer in the same way as nickel.

To obtain quantitative data on the specific influence of the alloying elements on the stability, a number of heats were chosen (Table I) and the M_{d30} temperature for each was determined as the characteristic measure of the stability of the austenite. In choosing the heats no attempt was made to vary one element at a time and to keep the others constant. On the contrary, all elements were allowed to vary simultaneously from one heat to another, and the values for composition and M_{d30} given in Table I were treated statistically by a multiple regression analysis.[24] In this way, the experimental work was kept to a minimum, and full use was made of the available material without loss of accuracy. The effect of the alloying elements on M_{d30} was assumed to be additive and to vary linearly with the percentage of the elements by weight, within the range studied. By statistical methods the following multiple regression equation was obtained:

$$M_{d30}(^\circ C.) = 413 - 462[(C + Ni)] - 9 \cdot 2[Si] - 8 \cdot 1[Mn] - 13 \cdot 7[Cr] - 9 \cdot 5[Ni] - 18 \cdot 5[Mo] \ldots (6)$$

where [Si], etc., is the weight percentage of the elements present. Carbon and nitrogen were taken together, as it is known that they are very similar in behaviour.[27]

The multiple correlation coefficient for the above relationship is 0·92, indicating that the assumptions made were justified. The significance of the individual coefficients was tested by means of the Student '*t*'-test. The probability of obtaining the above

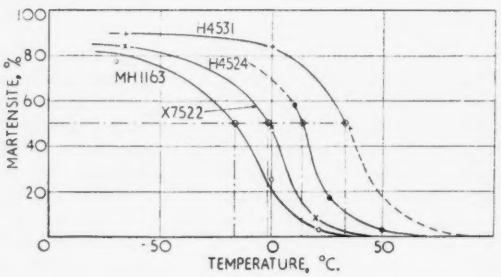

Fig. 10—Transformation/temperature curves at a true strain of 0·30, demonstrating evaluation of M_{d30} for 4 heats

(a) × 5000 (b) × 12,000

Fig. 11—Electron micrographs of an 18/8 stainless steel, deformed in tension to a true strain of 0.23 at 10° C., giving 16% of martensite

(a) × 7800 (b) × 12,000

Fig. 12—Electron micrographs of an 18/8 stainless steel deformed in tension to a true strain of 0.144 at −188° C., giving 62% of martensite

Fig. 13—Electron micrograph of an 18/8 stainless steel deformed in tension to a true strain of 0.235 at −30° C., giving 61% of martensite × 7800

Fig. 14—Electron micrograph of an 18/8 stainless steel deformed in tension to a true strain of 0.231 at −188° C., giving 83% of martensite × 7800

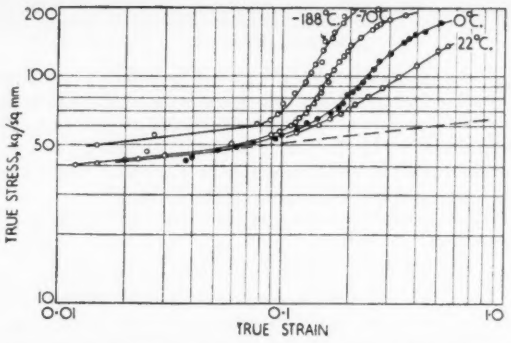

Fig. 15—True-stress/true-strain curves, showing influence of temperature. The curve for 0° C. is extrapolated to indicate the course of the curve in the absence of martensite formation

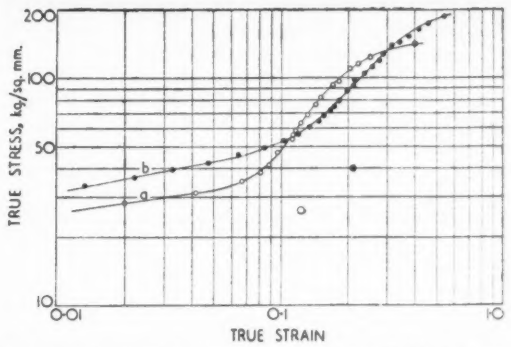

Fig. 17—Stress/strain curves for (*a*) heat *H*4531, of lower carbon content (0·06% C) and lower stability than (*b*) heat *E*1473 (0·16% C)

values of the coefficients by chance is $< 0{\cdot}001$ for (C + N), 0·01–0·001 for Mn, 0·02–0·01 for Cr, and $> 0{\cdot}05$ for Mo, Si, and Ni; *i.e.*, the results, according to commonly accepted standards, are clearly significant for carbon, nitrogen, and manganese, just significant for chromium, and not significant for molybdenum, silicon, and nickel. The results found for the last three elements are probably due either to the small amounts of silicon and molybdenum present or to the fact that these three elements varied only within a narrow range. The relative effect of the elements is often expressed in terms of their nickel equivalents. As the coefficient for nickel is insignificant the nickel equivalents were not calculated. Expressed as carbon equivalents, however, the results are consistent with those obtained by Eichelman and Hull in their investigation of the M_s point in 18/8-type stainless steels. The results also confirmed those of Eichelman and Hull in that, contrary to the views often encountered, the stability of the austenite was found to be increased by all the elements investigated.

By extending the range of the experimental work and by taking into account the interaction between the elements, it should be possible to obtain significant results even for nickel, molybdenum, and silicon.

A comparison of the experimental values of M_{d30} with the values as calculated from equation (6) is given in Table I. The standard deviation is $\pm 8°$ C. The possible error in equation (6) arising from chemical analysis is fairly large; an uncertainty of 0·015% in the carbon and nitrogen determination alone leads to an uncertainty of $\pm 6°$ C. in the calculated M_{d30} value. It is therefore felt that a standard deviation of $\pm 8°$ C. is the best that can be achieved in practice.

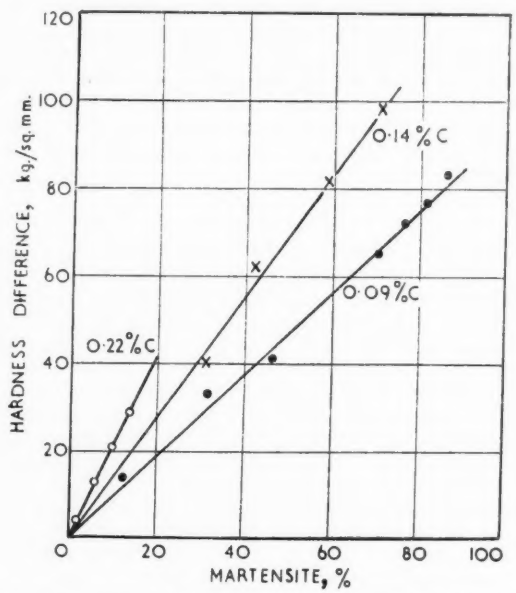

Fig. 16—Increase in hardness due to martensite formation at different carbon contents

Influence of Transformation on Mechanical Properties

The values for true stress and true strain obtained from the tensile specimens are plotted on a log/log scale in Fig. 15. For ordinary steels a straight line is obtained in such a diagram. This is not the case, however, with the austenitic steels studied in this work. Initially the curve is rectilinear and only a small increase in stress (hardness), due to the ordinary work hardening of the austenite, is observed. With further straining an abnormal increase in hardness occurs. This deviation was attributed to the formation of martensite, which does not deform as easily as austenite.

Taking the curve at 0° C. as an example, the initial rectilinear portion has been extrapolated to higher strains, to indicate the course of the curve in the absence of transformation. The difference in stress between the experimental and the extrapolated curves is the increase in hardness ascribed to the formation of martensite. A plot of this increase in hardness against the corresponding value of the martensite content (Fig. 16) shows a direct proportionality between these quantities, at least at the beginning of transformation. At higher strains and martensite contents deviation from proportionality occurs, owing to deformation and strain hardening of the martensite itself.

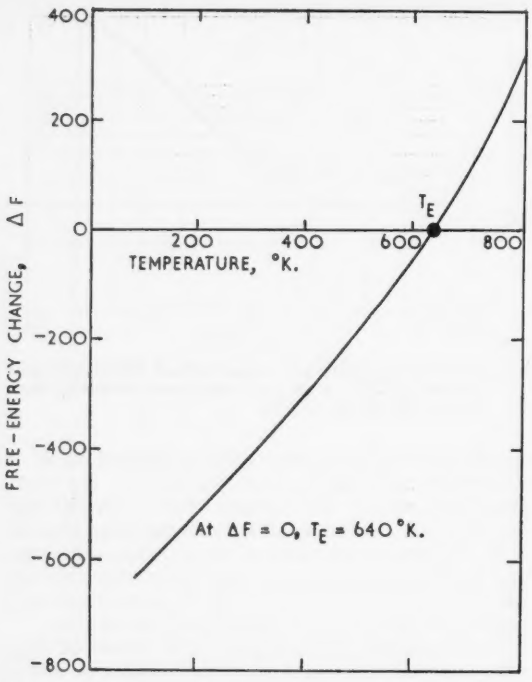

Fig. 18—Free-energy change attending austenite–martensite reaction in heat *X7522*

The curves in Fig. 16 are plotted for steels of three different carbon contents. As would be expected, the increase in hardness per 1% increase of martensite is greater the higher the carbon content.

Figure 17 illustrates the relationship between austenite stability, martensite formation, and carbon content. Heat *H*4531 has a lower initial hardness and a lower carbon content (0·06% C) than heat *E*1473 (0·16% C). Because of the lower stability of *H*4531, martensite formation starts at a lower strain in this alloy, and after some deformation it has become harder than *E*1473. The latter, however, gradually starts to transform and, as its martensite has a higher carbon content and is therefore harder, it again becomes the harder alloy. At the second point of intersection, at which they have the same hardness, heat *E*1473 contains 51% of martensite and *H*4531 contains 83%.

Calculation of Equilibrium Temperature T_E

The free-energy change ΔF (cal./g. atom) accompanying the martensitic transformation is given by Fisher[29] as:

$$\Delta F = N_{Fe}\Delta F_{Fe} + N_c(10{,}500 - 3{\cdot}425\,T) + \Delta F_* + \Sigma N_i K_i \quad \ldots\ldots(7)$$

where N_{Fe} = mol fraction of iron
N_c = mol fraction of carbon
N_i = mol fraction of alloying element
ΔF_{Fe} = free-energy change accompanying transformation of austenite to ferrite in pure iron
ΔF_* = free-energy change due to ordering of carbon atoms in tetragonal martensite
K_i = difference between the heat of solution of an alloying element in ferrite and in austenite.

Jones and Pumphrey[31] give K_{Ni} and K_{Mn} as 2500 and 2700 cal./g. atom, respectively, and introduce an empirical factor 1·25 in the first term of equation (7). Modified by this factor, equation (7) has been used successfully for iron–carbon–nickel alloys.[4] The value of K_{Cr} is estimated to be 1200 cal./mol.[30]

After the introduction of these values equation (7) becomes:

$$\Delta F = 1{\cdot}25N_{Fe}\Delta F_{Fe} + N_c(10{,}500 - 3{\cdot}425\,T) + \Delta F_* + 2700N_{Mn} + 1200\,N_{Cr} + 2500\,N_{Ni} \quad \ldots\ldots(8)$$

Using equation (8), ΔF was calculated for heat *X*7522; the influence of silicon, molybdenum, and nitrogen was neglected, because of the lack of thermodynamical data. In view of the small amounts of these elements present and the approximate nature of the calculation, the error due to this approximation was considered to be relatively unimportant. The results are plotted in Fig. 18. The intercept at $\Delta F = 0$ corresponds to the equilibrium temperature T_E. This was found to be about 640° K. (367° C.).

The T_E and M_d Temperatures

Figure 14 shows that at a strain of 0·30 no transformation occurs above 40° C. for heat *X*7522. A test bar pulled to a strain of 0·50, which is about the maximum strain obtainable in tension, gave no martensite at 80° C., and it was concluded that M_d is about 80° C. for this alloy. The value calculated for T_E was 367° C.; it does not seem probable, therefore, that the two temperatures can be identified.

A hypothetical explanation of the difference between the two temperatures is that with increasing temperature the critical resolved shear stress for slip decreases rapidly, whereas the stress for martensite formation increases. At the same time the probability of thermal relaxation of stresses in the lattice and of the strain embryos for the martensitic reaction greatly increases. At sufficiently high temperatures it is possible, therefore, that even below T_E the stress necessary for martensite formation is never reached, or is reached only after the retarding forces have become too great.

DISCUSSION

The experimental results will now be discussed in the light of the ' reaction-path theory ' formulated by Cohen. As mentioned in the introduction, the free-energy change has to be large enough for the reaction to mount the activation barrier between the austenitic and the martensitic states. For the spontaneous reaction this happens at the M_s temperature. Above M_s but below T_E there is a supply of free energy available, which, however, is insufficient to initiate transformation. The additional energy required may be supplied as mechanical energy, *e.g.*, by uni-axial tension of the test bar, whereby an external stress field is superimposed on the local stress fields that constitute the embryos. In this way some embryos may reach the energy state necessary to carry them over the activation hump, thus starting transformation. The work U done on or by the transformation,

as the acting forces are carried through the transformation displacements, can be written:[4]

$$U = \tfrac{1}{2}[\gamma_0 \sin 2\theta + \epsilon_0(1 + \cos 2\theta)]\sigma_1 \quad \ldots\ldots\ldots (9)$$

where γ_0 = shear strain of the transformation along the habit plane
ϵ_0 = dilatational strain normal to the habit plane
θ = angle between the axis of the tensile specimen and the normal to the habit plane
σ_1 = external tensile stress.

It is natural to assume that in a polycrystalline specimen the embryos are statistically oriented and distributed. It follows that the first martensite platelets to form have their habit plane at an angle θ yielding the maximum value of U. As the values for γ_0 and ε_0 are known to be approximately 0·20 and 0·04 respectively, θ may be calculated; for an iron–nickel alloy it was found that $2\theta = 79°$.[4] Accordingly, the first embryos to become activated should be the most energetic or those most favourably oriented, *i.e.*, having their potential habit planes favourably oriented in relation to the external stress field.

As the chemical free-energy change ΔF decreases with increasing temperature, the mechanical work U to be added should show a corresponding increase. This has been confirmed experimentally (Figs. 5 and 8). The increase is, however, generally larger than is justified from the thermodynamical point of view, and is evidence that at higher temperatures a large proportion of the deformation energy is consumed by slip. Thus, the energy required for the formation of the first 1% of martensite (as obtained by extrapolation of the experimental curves in Fig. 6 to zero martensite content) is increased by 6·5 cal., or 650 cal. per mol of 100% martensite, as the temperature is raised from −30° to 0°C., whereas the theoretical value of the energy difference (obtainable from Fig. 18) for this temperature interval is only 31 cal. The corresponding measured and calculated values for the temperature interval of −188° to −70°C. are 90 and 132 cal., respectively. The difference of 42 cal. between the last two figures probably lies within the limits of error. It is concluded that at − 188° C. the critical shear stress for slip is so high that practically no slip occurs and deformation takes place entirely by the martensitic mode. This is also supported by the microscopical evidence.

It may seem strange that large plastic strains could be obtained with no slip occurring; this might be explained schematically as follows. In the spontaneous transformation of austenite to martensite the only externally observable effect is that of the dilatation ($\varepsilon_0 = 0{\cdot}04$); the shear displacements ($\gamma_0 = 0{\cdot}20$) are statistically oriented with no resultant external change of shape. Under the influence of a uni-axial tension, transformation is forced to take place in strongly preferred directions, and consequently, with a large number of elementary shear displacements taking place in approximately the same direction, the net result will be an external change of shape.

From equation (9) it might be concluded that the formation of martensite is a function of stress alone, the latter increasing with increasing temperature, but this is true only to a limited extent. In a polycrystalline material the applied external stress is only a measure of the average stress in the material over the cross-section of the test bar. Owing to the plastic straining of the material, peaks of stress above the average stress level are set up in the lattice. Some of these peaks may be high enough to nucleate some martensite before the average stress has reached the level necessary for martensite formation. Through the work-hardening action of the strain the stress may thus be brought up to the critical level in some places. It may then be said that strain has as important a rôle as has stress. But it can also be said that strain is only a consequence of the martensitic mode of deformation, just as it is a consequence of the slip mode of deformation, taking place simultaneously and independently. There is indeed a complex interplay between cause and effect, but it is felt that the application of energy considerations does clarify the picture somewhat.

The anomalous behaviour of the curve for − 188° C. in Fig. 4 may now be easier to understand. At this temperature only slight yielding or none occurs before the average stress has reached the critical stress for martensite transformation. At higher temperatures the critical stress for martensite formation is higher but, because of the slip now occurring, local stress peaks may initiate transformation at a general stress level lower than that required at − 188° C.

During the early stages of transformation a stimulating effect was observed, as demonstrated by Figs. 3 and 6 and also by the log-autocatalytic form of equation (5). Cohen[8] reports a similar observation in a 30% Ni–70% Fe alloy and attributes it to the formation of new embryos, as an autocatalytic effect of the transformation.[32]

It is assumed that, as a result of slip and martensite formation, new embryos are formed and stresses are set up in the lattice. These stresses add so much energy to some embryos that they become supercritical and nucleate martensite. Statistically, over a sufficiently large volume, as many embryos are hindered as are aided by the stresses. Initially, however, the aiding effect is more noticeable, as it leads to premature martensite formation before the average stress has reached its critical value; a stimulating effect is therefore observed. At a later stage, when the most energetic or the most favourably oriented embryos have been consumed, more energy is required to activate embryos of successively less favourable orientation and less energy, and the external stress has therefore to be increased. The opposing effect gradually becomes more noticeable as it decreases the average energy level of the remaining embryos, and a stabilizing effect is now observed. Simultaneously, the amount of martensite formed per nucleus gradually decreases, owing to the partitioning of the austenite into smaller and smaller volumes by the martensite platelets that are formed. Consequently, the surface energy per mol of martensite increases. Finally, the opposing effects become larger than the stimulating effects of the external stress, and the transformation gradually peters out. Even under the most favourable conditions for martensite formation, at − 188° C., the opposing effects are so large that transformation can never be complete, but stops at a limiting value M_T of about 90% of martensite.

The amount of slip per 1% of martensite increases with the temperature; this is illustrated by Fig. 6, where the minimum in the curves is narrower and displaced towards lower percentages of transformation the higher the temperature, and by Fig. 2, where owing to this increasing amount of slip the transformation stops at a lower value of M_T with increasing temperature. On thermodynamical considerations alone M_T would have the same value at all temperatures below T_E.

CONCLUSIONS

(1) The formation of martensite as a function of plastic strain, stress, and deformation energy has been determined in a tensile test for a number of austenitic stainless steels. The results are in accord with Cohen's reaction-path theory of the martensitic transformation.

(2) The deformation of martensite is regarded as a mode of deformation competing with the slip mode of deformation.

(3) The governing factor in the formation of martensite induced by deformation is considered to be the supply of mechanical energy through deformation. Stress is of primary importance here, as deformation is shown to be only a consequence of the formation of martensite caused by the action of stress. However, strain, either as the result of the martensite or of the slip mode of deformation, is assumed to be of some importance, as it affects the stress conditions so that local peaks of stress may be formed, activating some martensite embryos.

(4) The influence of temperature is discussed in terms of the relation between the martensitic and the slip mode of deformation. At very low temperatures it was found that the martensitic mode is the dominating if not the only mode of deformation. At higher temperatures below M_d slip becomes the prevalent process of deformation.

(5) Within a narrow temperature range, a radical change of stability of the austenite was observed, and was attributed to a transition between the two modes of deformation. In this range a characteristic temperature, M_{d30}, was chosen as a measure of the stability of the austenite that is easier to determine practically than M_d.

(6) The effect of composition on the austenite stability, measured as the M_{d30} temperature, was evaluated by means of a multiple regression analysis, applied to several heats of varying composition. Contrary to the views often expressed in the literature, it was found that chromium, silicon, and molybdenum act as stabilizers of austenite in the martensitic transformation in the same way as nickel, manganese, carbon, and nitrogen. The results largely confirmed Eichelman and Hull's investigation of austenite stability using another measure of the stability.

(7) Finally, the anomalous increase in stress observed in the stress/strain curve was treated quantitatively, and was found to be proportional to the amount of martensite formed in the tension test and also to the carbon content of the martensite.

Acknowledgments

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A Gravimetric Method of Gas Analysis

By J. Morsing

SYNOPSIS

A gravimetric method for the simultaneous determination of hydrogen sulphide, carbon dioxide, oxygen, unsaturated hydrocarbons, hydrogen, carbon monoxide, methane, ethane, and nitrogen (by difference) in fuel gases is outlined. The apparatus used is described, and details for carrying out the analysis are given. Two or three analyses may be made per day with one apparatus. The method is especially suited for analysing producer gas, but with the appropriate absorbents other gaseous mixtures may be similarly analysed. 873

Introduction

VOLUMETRIC gas analyses carried out with different apparatus or by different persons often give widely differing results. The aim of the investigation described in this paper was to improve the methods of technical gas analysis and if possible to establish a standardized procedure for the Swedish steel industry, especially for the analysis of producer gas.

A study of the most common causes of error in volumetric methods of gas analysis indicates clearly the desirability of an adequate method of control of such analyses. Attempts were therefore made to analyse gas mixtures gravimetrically by the successive absorption and weighing of the different gas components in a sample.

The main advantage of a gravimetric method is that solid absorption agents may be used and that the errors caused by the physical solubility of the gas components in the absorbents are eliminated. In addition, errors due to changes in temperature and pressure during the analysis, or to the influence of the draining time on the burette readings, are also avoided.

Gravimetric determinations of single gas components have been carried out by several investigators, as a check on certain volumetric methods of gas analysis. Thus, Terres and Mauguin[1] gravimetrically followed the fractionated combustion over copper oxide according to Jäger,[2] and gravimetric determinations of hydrogen, carbon monoxide, and methane have been carried out by Ott,[3] Scherf,[4] and Schmidt.[5] Ahrnbom[6] used gravimetric methods for the determination of the total hydrogen and carbon contents in a special investigation of the gas obtained by distillation of bituminous shale.

A method for gravimetric determination of carbon dioxide and carbon monoxide, in gas mixtures containing only these gases and nitrogen, was recently published by Toensing and McKinney.[7] These workers absorbed and weighed the CO_2, converted the CO by combustion over copper oxide, and absorbed and weighed the CO_2 formed.

These earlier investigations have shown that gravimetric methods may be used with advantage for the determination of carbon dioxide, carbon monoxide, and hydrogen in gas mixtures which do not contain hydrocarbons.

The present author's aim was to work out a practicable gravimetric method for the complete analysis of producer gas, including the determination of hydrogen sulphide, carbon dioxide, oxygen, heavy hydrocarbons, hydrogen, carbon monoxide, methane, and ethane. The experiments have led to the method described in this paper, which seems well suited for the analysis of technical gases, particularly producer gas, with the aid of a good analytical balance; a microbalance is not necessary.

PRINCIPLE OF THE METHOD

A certain volume of gas (100 or 200 ml.), either saturated with moisture or fully dried, is accurately measured in a burette. After the temperature and pressure have been recorded, the measured gas sample passes through a train of absorption tubes for water, hydrogen sulphide, carbon dioxide, oxygen, and unsaturated hydrocarbons, a combustion tube containing copper oxide heated to about 290° C. for the fractional combustion of hydrogen and carbon monoxide, tubes for the absorption of the water and carbon dioxide formed in the combustion, an additional combustion tube containing a mixture of copper oxide and iron oxide heated to about 650° C. for the combustion of methane by the method of Arneil,[8] and finally tubes for the absorption of the resulting water and carbon monoxide.

At the start of the analysis the whole apparatus, including the absorption tubes, must be flushed with nitrogen and the absorption tubes weighed. After the entire gas sample has been displaced from the burette and passed through the absorption train, the apparatus is flushed with pure nitrogen and the absorption tubes, filled with nitrogen, are disconnected and weighed. From the increase in the weight of the absorption tubes the volumes at N.T.P. of the gas components are calculated. The measured volume of the gas sample is also reduced to N.T.P. and corrected for moisture content, and the percentages by volume of the components are calculated on a dry basis. Nitrogen must be used to flush out the train if the percentage of oxygen is to be determined. In this case, the copper oxide in the combustion tubes is gradually reduced, and the copper formed must occasionally be reoxidized by suction of dry oxygen

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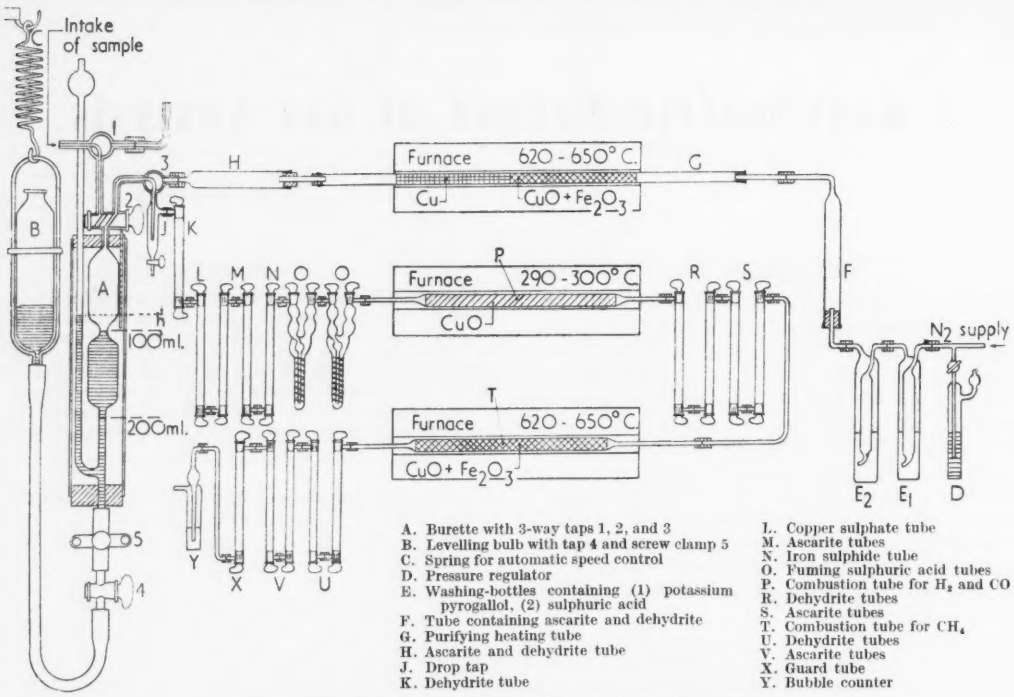
Intake of sample
Furnace 620 - 650° C.
Cu
CuO + Fe2O3
Furnace 290 - 300° C.
CuO
Furnace 620 - 650° C.
CuO + Fe2O3
N2 supply
100 ml.
200 ml.
A. Burette with 3-way taps 1, 2, and 3
B. Levelling bulb with tap 4 and screw clamp 5
C. Spring for automatic speed control
D. Pressure regulator
E. Washing-bottles containing (1) potassium pyrogallol, (2) sulphuric acid
F. Tube containing ascarite and dehydrite
G. Purifying heating tube
H. Ascarite and dehydrite tube
J. Drop tap
K. Dehydrite tube
L. Copper sulphate tube
M. Ascarite tubes
N. Iron sulphide tube
O. Fuming sulphuric acid tubes
P. Combustion tube for H2 and CO
R. Dehydrite tubes
S. Ascarite tubes
T. Combustion tube for CH4
U. Dehydrite tubes
V. Ascarite tubes
X. Guard tube
Y. Bubble counter

Fig. 1—Diagram of gravimetric gas-analysis apparatus

or air through the combustion tubes at about 400° C. If it is not necessary to determine the oxygen content it is preferable to flush the apparatus with pure oxygen instead of nitrogen, as regeneration of copper oxide is then unnecessary.

Anhydrous copper sulphate dried at 150–160° C. is used for the absorption of hydrogen sulphide (Fresenius[9]), and ascarite (soda asbestos) and dehydrite (anhydrous magnesium perchlorate) are used for the absorption of carbon dioxide and water. The absorption of oxygen is effected by iron sulphide, as proposed by Griffith[10] and successfully used by Gooderham.[11] The iron sulphide is prepared in the absorption tube by passing hydrogen sulphide over dried iron hydroxide, with which the tube has previously been filled.

These reagents have all proved very satisfactory as solid absorbents for the particular gas components. On the other hand, it has not been possible to find any appropriate solid reagent for the absorption of the unsaturated hydrocarbons, which should be removed before the fractional combustion of hydrogen and carbon monoxide. Several attempts were made to adsorb the unsaturated hydrocarbons in a column of active carbon or of silica gel, but it was difficult to adjust the time so that the other components were completely removed before the nitrogen stream began to carry away the unsaturated hydrocarbons. Turkeltaub[12] has shown that the unsaturated hydrocarbons can be completely removed in a column of active carbon, but even if this procedure could be used it would be very troublesome.

Several unsuccessful attempts were made to absorb the unsaturated hydrocarbons by sulphuric acid activated by various added agents, such as uranyl sulphate,[13] nickel and silver sulphate,[14] and mercuric sulphate,[15] using different designs of absorption tubes. Finally, the classic absorbent, fuming sulphuric acid containing about 20% of free sulphur trioxide, was tried. It might be thought that this reagent, which easily gives off fumes of sulphuric acid, cannot be used without loss of SO_3. On passing a dry gas through the acid in the specially designed absorption tube, however, no mist or fumes could be observed on the surface of the acid, and the absorption tubes, with guard tubes containing ascarite and dehydrite, showed no change in weight in blank experiments. With the tube design described below, a single sulphuric acid tube proved to be sufficient in the analysis of producer gas and town gas containing up to 3% of unsaturated hydrocarbons. For the analysis of gases with higher hydrocarbon contents, additional absorption tubes may be used.

APPARATUS

Figure 1 shows the arrangement of the apparatus developed for the gravimetric analysis of a gaseous mixture of hydrogen sulphide, carbon dioxide, oxygen, unsaturated hydrocarbons, hydrogen, carbon monoxide, methane, ethane, and nitrogen (as residue).

Measurement of Gas Sample

The bulb burette *A* is connected at its upper end with the three-way stop-cocks 1, 2, and 3, and at the lower end, through a rubber tube and stop-cock 4, with the levelling bulb *B*; mercury is used as confining liquid. The levelling bulb may hang from the spring *C* for automatic regulation of the gas velocity during analysis.[16]

The burette is calibrated below tap 2 for the accurate measurement of 100 and 200 ml.; it is enclosed in a water jacket, together with a manometer tube and a thermometer. The mercury meniscus may be accurately adjusted, after closing tap 4, by means of the screw clamp 5 on the rubber tube between tap 4 and the burette. Pure mercury is poured into B until A is filled to tap 1. The position of B should be such that it then contains about 5 ml. of mercury.

Tap 1 is used for introducing the sample, and for flushing the connecting tube between the container for the gas sample and the gas-analysis apparatus. By means of taps 2 and 3 the burette may be connected either to the absorption and combustion train or to the purification train for nitrogen.

Nitrogen Purification Train

The arrangement for the purification of nitrogen consists of the following parts (Fig. 1):

A pressure regulator D for regulating the velocity of the nitrogen, which flows directly from the high-pressure container

Two washing bottles E_1 and E_2 containing potassium pyrogallol and sulphuric acid, respectively; the latter is used for observing the bubbling velocity of the gas

An absorption tube F filled to about one-third with ascarite and two-thirds with dehydrite

A combustion tube G heated to 650° C. and filled to about one-third with a mixture of 96% copper oxide and 4% iron oxide for the combustion of impurities, and two-thirds with metallic copper chips for the absorption of oxygen*

An absorption tube H with ascarite and dehydrite to absorb carbon dioxide and water.

Apparatus for Absorption and Combustion

The purified nitrogen and the gas sample pass through tap 3 to the absorption and combustion train, which consists of the following parts (Fig. 1):

The drop trap J, to retain mercury drops which may pass through tap 3

The drying tube K, containing dehydrite

The absorption tube L, containing anhydrous copper sulphate for the absorption of H_2S. This tube may be omitted when gases very low in sulphur are analysed. Any H_2S present is then absorbed together with CO_2, as is generally the case in volumetric gas analysis

One or two absorption tubes M, containing ascarite and dehydrite for the absorption of CO_2

The absorption tube N, containing ferrous sulphide and dehydrite for the absorption of O_2

One or two specially constructed absorption tubes O, for the absorption of unsaturated hydrocarbons with fuming sulphuric acid

The combustion tube P, containing copper oxide and heated in an electric furnace to 290° C. for the fractional combustion of H_2 and CO

One or two absorption tubes R, containing dehydrite for the absorption of H_2O corresponding to the gas content of H_2

One or two absorption tubes S, containing ascarite for the absorption of CO_2 corresponding to the gas content of CO

The combustion tube T, heated to 650° C. and containing a mixture of 96% copper oxide and 4% iron oxide prepared by Arneil's method[8] for the combustion of CH_4

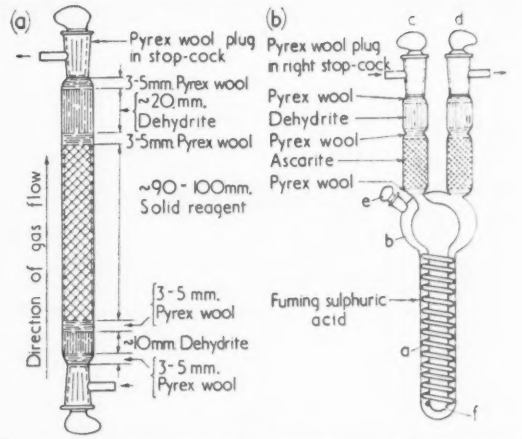

Fig. 2—Absorption tubes: (*a*) containing solid reagents, for H_2S, CO_2, or O_2; (*b*) for unsaturated hydrocarbons

One or two absorption tubes U, containing dehydrite for the absorption of H_2O corresponding to the gas content of CH_4

One or two absorption tubes V, containing ascarite for the absorption of CO_2 corresponding to the gas content of CH_4

The guard tube X, containing dehydrite and ascarite in reverse order to that in the absorption tubes, to guard against the entry of moist air into tube V

The bubble counter Y.

Absorption Tubes

The absorption tubes are made of soda glass; tubes of Pyrex or Jena glass are unsuitable, as they are easily charged with static electricity in dry weather and need a long time to be discharged and to assume a constant weight. For the solid absorbents straight tubes are used, of about 10-ml. capacity, 12-mm. dia., and about 200-mm. total length (*see* Fig. 2*a*). The tubes have precision-ground glass stop-cocks, the side tubes of which are 10 mm. long with external and internal diameters of about 4 mm. and 2 mm. respectively. The side tubes have ground ends and must be quite straight, as they must be fitted together with glass-to-glass contact by small (12-mm. long) pieces of vacuum rubber tubing. Such joints were found to be completely gas-tight, and the errors arising from the solubility of the gas components in rubber are negligible.[17]

New tubes are first cleaned in the ordinary way with bichromate–sulphuric acid, and then washed in water and alcohol and dried at 100° C. In refilling the tubes the waste reagents are first emptied out, the stop-cocks are degreased with ether, and the tubes are washed with water and alcohol and dried at 100° C.

In filling the tubes a small funnel of thin nickel sheet should be used, to prevent the reagents from coming in contact with the ground surfaces of the stop-cocks or from penetrating into the side tubes.

For tubes that are to be filled with solid absorbents a plug of Pyrex wool about 5 mm. thick is first placed in the tube and the reagents are then introduced (Fig. 2*a*). For the dehydrite tubes only dehydrite is used. Finally, a second plug of Pyrex wool about

* The copper–iron oxide mixture is prepared (Arneil[8]) by mixing 96 g. of copper oxide with 20 g. of crystallized ferric nitrate dissolved in a little water, evaporating the mixture to dryness, and igniting at 700–750° C. to decompose the nitrate.

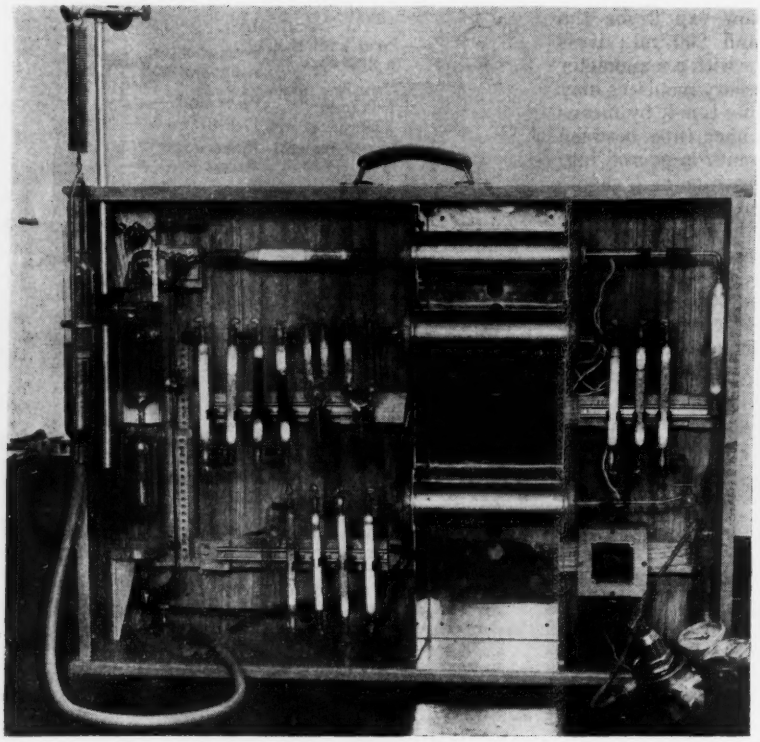

Fig. 3—Portable apparatus for gravimetric gas analysis

5 mm. thick is inserted. To indicate the direction of the gas stream it is advisable to insert a little Pyrex wool into the exit stop-cock. It is very important that the gas should always pass in the same direction through the tubes. If, for instance, a dehydrite tube is used in the reverse direction to that generally used, part of the water absorbed in a previous analysis will pass to the next absorption tube. The first dehydrite layer in the absorption tubes (Fig. 2*a*) is intended to prevent moisture from escaping backwards from the tube in case the flow of gas should be stopped or reversed.

To lubricate the stop-cocks the ground surfaces are first carefully cleaned with ether and dried. A very thin layer of Vaseline is rubbed on the surface of the tap, which is inserted in the tube and turned once or twice, and then taken out and well dried. With a finger a thin streak of 'special cock grease'[19] is applied to the side of the tap opposite the bore-hole and the tap is inserted without turning until the grease is distributed around the tap. Finally, the tap is turned and the grinding must then appear completely glass-clear with no stripes or bubbles of air. If this is not the case, the lubrication must be repeated. The side tubes are very carefully cleaned with a pipe cleaner moistened with ether and alcohol. The outsides of the tubes are rinsed with distilled water and lightly rubbed with a moist towel.

The absorption tube containing fuming sulphuric acid for the absorption of unsaturated hydrocarbons is seen in Fig. 2*b*. It consists of a gas-washing tube *a*, the upper part of which is widened to a bulb *b*, with inlet and outlet guard tubes *c* and *d* and a side tube *e* for filling and emptying. The guard tubes should contain ascarite in the lower part and dehydrite in the upper. From *c* an inner thin-walled glass tube (about 8-mm. dia.)leads to the bottom of the outer tube (about 18-mm. dia.). This tube is also widened to a bulb in the upper part, as a trap for the acid should back-suction occur; at its lower end it has a fine opening *f* (about 1 mm.). In the space between the inner and the outer tube a loose spiral of low pitch is inserted, made of thin glass tubing and sealed by fusing in both ends. When the gas is passed, fine bubbles emerge from the opening *f* and slowly rise along the spiral. When the tubes are prepared for analysis they are packed with ascarite and dehydrite between plugs of Pyrex wool as in the other tubes. The fuming sulphuric acid is introduced through side-tube *e* with a pipette and care is taken that the ground surface is not contaminated by the acid. The stopper is inserted, care being taken to see that it is gas-tight, and the outside of the tube is rinsed with distilled water and lightly rubbed with a moist towel.

In weighing, the absorption tubes are tared against tubes of the same shape and approximately the same weight. For the tubes containing solid absorption agents a common tare tube may be used; this is charged with pumice or crushed quartz between plugs of Pyrex wool so that it has approximately the same weight as the absorption tubes. As tare for the sulphuric acid tube a similar tube with the same filling is used. The stop-cocks of the tare tubes are lubricated and the tubes are cleaned in the same way as the main tubes.

Electric Furnaces and Combustion Tubes

The electric furnaces for heating the combustion tubes are of 18-mm. internal dia. and 220-mm. length. The inner part of each furnace consists of a tube of heat-resisting steel surrounded by Alundum cement in which a resistance spiral of Kanthal wire is embedded. The steel tube distributes the heat so that a uniform temperature is obtained throughout most of the length of the furnace. For regulating the temperature a 'laboratory transformer,' with tappings for arbitrary voltages up to 200 V., is suitable. The same transformer may be used for all three furnaces and, with suitable regulation of the voltage, the tempera-

ture in the furnaces may be kept sufficiently constant without the use of rheostats. Thermo-elements are inserted in the furnaces, for measuring the temperature.

The combustion tubes are made of Supremax glass and have an external dia. of about 15 mm. They are filled to a length of about 160 mm. with copper oxide or a mixture of copper oxide and ferric oxide, which is packed between plugs of quartz wool and inserted so that the whole oxide filling is within that part of the furnace which is at constant temperature.

Assembly of the Apparatus

The three furnaces are placed above one another in a stand of aluminium sheet (Fig. 3), which serves to guard the absorption tubes from direct radiation from the furnaces.

On both sides of the furnaces there are specially designed holders for the absorption tubes. These consist of elastic clamps of nickel-plated phosphor-bronze with **T**-shaped feet, which glide easily in a horizontal guide of nickel-plated brass. As the tubes are inserted vertically and are readily movable in the clamps, they may easily be adjusted in the apparatus both horizontally and vertically and may thus be fixed in suitable positions without the use of screw clamps.

The whole apparatus may be housed in a portable wooden case in which the gas burette and the levelling bulb are placed on one side and the nitrogen gas inlet on the other (Fig. 3).

ANALYTICAL PROCEDURE

The analysis may be made either on a gas sample previously dried over dehydrite (magnesium perchlorate) or on one saturated with moisture. In the former case the measuring burette and the mercury must be completely dry and part of the gas sample must be used for flushing the drying tube. It is therefore preferable to use a gas sample saturated with moisture, especially if only a limited amount of gas is available (*e.g.*, 250 ml. in a gas-sampling pipette). For the analysis of a gas containing H_2S, however, the gas sample must be previously dried.

Preparation of the Apparatus for Use

The three electric furnaces are heated to the prescribed temperatures and nitrogen from a gas container is passed through the purifying train for some minutes at about 30 ml./min. (*i.e.*, about 2 bubbles per second in the wash-bottle E_2). The velocity may be regulated by the pressure regulator D. The nitrogen leaves the purifying train by tap 3 and drying tube K.

The absorption tubes are connected in order, beginning with the copper sulphate tube L, in a direction such that the tap marked with glass wool is always at the exit end of the tube. Pieces of good rubber tubing 12 mm. long, about 3 mm. internal dia., and 2 mm. thick are used as connections; when these are not in use they are stored in a small closed box. The nitrogen is allowed to pass continuously through the tubes as they are connected. The whole train is tested for gas-tightness as each tube is connected, by closing the exit tap of the last tube; the bubbling in wash-bottle E_2 should then cease.

The whole system of absorption and combustion tubes should be flushed with nitrogen for at least ½ hr. after all the tubes have been connected. The stop-cocks of the guard-tube X are closed, and as soon as the pressure in the system is equalized the stop-cocks of all the absorption tubes, going backwards from V to L, and of the drying tube K, are closed. A slight excess pressure then obtains in all tubes.

The absorption tubes are then removed from the apparatus, wiped lightly with a clean moist towel, and placed together with the tare tubes on a rack of aluminium sheet near the analytical balance. Immediately the tubes nearest to the combustion tubes are disconnected, the ends of the latter should be closed with small glass plugs to prevent moist air from entering. The absorption tubes are weighed after about 15–20 min. as described below.

Introduction of Gas Sample

Taps 1 and 2 are turned to connect the burette A with the atmosphere. Tap 4 and clamp 5 are opened and the levelling bulb B is raised until the mercury in A reaches tap 2. Tap 2 is turned to connect A with tap 3, and 20–30 ml. of purified nitrogen are introduced into the burette through taps 3 and 2 to flush out the air in the capillary between 3 and 2; the nitrogen is removed through 2 and 1. The burette wall is moistened with water by turning the capillary attached to tap 1 through 180°, dipping it into a beaker of distilled water, and lowering the mercury level to the lower mark on the burette. A few ml. of water are thus drawn into the burette. Bulb B is raised until the mercury again reaches tap 1; a sufficient amount of water then remains on the burette wall to saturate the gas to be analysed. The levelling bulb B is suspended from the spring C and its position regulated so that the mercury level in A is at tap 2 when the connection between A and B is open.

Tap 1 is turned to connect A with the sample container, and about 20 ml. of the gas sample are introduced into A by lowering B. This first quantity of the sample is contaminated by air from the capillary between 1 and the container, and is expelled through the right-hand branch of tap 1.

A sample for analysis may now be taken into the burette by connecting A with the container through 1 and lowering B until the mercury in A almost reaches the calibration mark for 100 or 200 ml. Taps 4 and 2 are closed and B is suspended from the spring C. The position of the meniscus in A is brought exactly to the mark intended, by means of the screw clamp 5. The level in the manometer tube may rise or sink, depending on the pressure in A. The apparatus is left for about 10 min. to permit temperature and pressure equilibrium to be established, and the temperature in the water jacket (t° C.), the atmospheric pressure (b mm. of Hg), and the difference in level between the mercury levels in B and A (h mm.) are then noted. The volume at N.T.P. of the dry gas sample may then be calculated as shown below.

Absorption and Combustion with Subsequent Absorption

After the gas sample in the burette has been measured, the weighed absorption vessels in the

Table I
SPECIFIC VOLUMES OF GASES

Gas	Formula	Density, mg./ml.	Specific Volume at N.T.P., ml./mg.
Hydrogen sulphide	H_2S	1·539	0·6497
Carbon dioxide	CO_2	1·9769	0·5058
Oxygen	O_2	1·42904	0·6997
Ethylene*	C_2H_4	1·2604	0·7933
Hydrogen	H_2	0·08988	11·126
Carbon monoxide	CO	1·2504	0·7997
Methane	CH_4	0·7168	1·395
Ethane	C_2H_6	1·3566	0·7371

* The total amount of heavy hydrocarbons in these analyses of producer gas is calculated as ethylene. For gas mixtures also containing heavier hydrocarbons the conversion factor must correspond to the density of the gas mixture; this may be computed if the proportion of the hydrocarbons is known.

absorption and combustion train are reconnected, beginning with tube *K*. A stream of purified nitrogen should pass through the tubes continuously and the rubber connections should be tested for gas-tightness as described above. When all the tubes have been connected, the gas sample is passed through the apparatus.

Clamp 5 is screwed tight, tap 4 is opened, and the mercury is then allowed to pass slowly from *B* to *A* by regulating with screw clamp 5 until an excess pressure of about 20 mm. of Hg is obtained in *A*. The latter is connected with the absorption and combustion system by taps 2 and 3 and the gas is allowed to stream from *A* through the apparatus at about 10–12 ml. per min. The velocity is regulated by the screw clamp 5 and then automatically by the spring *C*. The mercury level in *A* is brought to tap 3, which is then turned immediately so that purified nitrogen again passes through the absorption and combustion system. Nitrogen is passed through the train for about 2 hr. at about 30 ml. per min. to flush out all remaining gas from the apparatus. The taps of all the absorption tubes are then closed successively, beginning from the end to obtain an excess pressure of nitrogen in them. The tubes are disconnected, wiped as described above, and put on the rack to be weighed.

Weighing of the Absorption Tubes

The tare tubes which are used in weighing should, during the analyses, be placed on the tube rack close to the apparatus. They are wiped with a clean, moist towel at the same time as the absorption vessels and placed together with these on the aluminium rack, which is then placed near the balance.

The excess pressure in the absorption tubes and in the tare tubes should be equalized with atmospheric pressure after about 15 min. by cautiously opening and immediately closing the exit tap (marked by Pyrex wool), and the tubes replaced on the rack. Only the tip of the tap should be handled, so that the tube is not heated by the hand. The entrance tap of the tube must not be opened, as some moisture absorbed by the tube might be lost. After being replaced on the rack the tubes should remain there for at least 5 min. before they are weighed.

The tubes are handled with a pair of tongs made of stainless-steel sheet and suspended on hangers of stainless-steel wire above the balance pans. The weight is read 2 min. after releasing the balance. Repeated trials have shown that the weights of the absorption vessels obtained in this way are constant over a long period, sometimes for several days if the stop-cocks are absolutely tight. It is therefore not necessary to prescribe a precise time after disconnecting the tubes from the apparatus for the weighing.

The tubes may also be weighed without being wiped with a moist towel each time, but in that case they will require a longer time to attain constant weight. The tubes are slightly cooled by wiping and their surfaces are saturated with moisture, and they therefore attain constant weight more quickly.

For weighing, a Sartorius air-damped analytical balance (200 mg. max. load) with a reading accuracy of 0·05 mg. was used. Repeated weighings of the absorption tubes, filled with pure nitrogen, have shown a reproducibility equal to the sensitivity of the balance.

To ensure that the absorption is complete two absorption tubes in series have been used for each component (Fig. 1). Generally, no increase in weight was obtained in the second tube. In most cases, therefore, it should be sufficient to have one absorption tube for each component.

CALCULATION OF RESULTS

Let the temperature of the gas sample (= temperature of water jacket) be $t°$ C., and the barometer reading be b mm. of Hg.

The pressure of the gas sample is shown by the difference in level h mm. between the mercury levels in *A* and *B*. The total pressure of the gas sample is thus $(b + h)$ or $(b - h)$ depending on whether the gas pressure is higher or lower than the atmospheric pressure. The total pressure at $t°$ C. is corrected for the thermal dilatation of the mercury and the scale used. This correction, for pressures between 740 and 760 mm. of Hg and temperatures between 20° and 22° C., varies from 2·4 to 2·7 mm. of Hg and should be deducted from the measured pressure level. If 2·5 mm. is deducted in each case, the relative correction error is not more than 0·03%; *i.e.*, the absolute error for a carbon dioxide content of 30% is equal to about 0·01%.

The dry gas pressure is obtained by subtracting the saturation pressure p_w at $t°$ C. from the total gas pressure. Thus, the pressure of the dry gas sample $p = b + h - p_w$ or $p = b - h - p_w$ mm. of Hg. The volume of the sample introduced for analysis (100 or 200 ml.), corrected for moisture content, is reduced to N.T.P. by multiplying it by

$$\frac{p}{760} \times \frac{273}{273 + t}.$$

The increase in weight of the absorption tubes due to the absorbed components, measured in mg., is converted to ml. of gas by multiplying by the specific volumes of the gases (*see* last column of Table I).

Hydrogen sulphide, carbon dioxide, oxygen, and heavy hydrocarbons are absorbed without change, and their proportions may be calculated directly from

Table II

EXAMPLE OF GRAVIMETRIC ANALYSIS RESULTS

Gas Component	Weights of Absorption Tubes			Conversion Factor, ml./mg.	Volume at N.T.P., ml.	Volume in Dry Gas, %
	Initial	Final	Increase			
CO_2	3·08925	3·09595	6·70	0·5058	3·39	3·47
O_2	0·50880	0·50900	0·20	0·6997	0·14	0·14
C_nH_m	0·50935	0·50960	0·25	0·7933	0·20	0·20
H_2	0·64965	0·66200	12·35	1·2448	15·37	15·74
CO: Tube 1	3·37365	3·42955	55·90	0·5090	28·45	29·13
Tube 2	3·81570	3·81570	0·00	0·5090	...	...
CH_4						
H_2O	0·03185	0·03460	2·75	0·6207	1·71	1·75
CO_2	2·49315	2·49640	3·25	0·5085	1·65	1·69

the increases in weight of the tubes. However, for hydrogen, carbon monoxide, methane, and ethane the increase in weight corresponds to the weight of water or carbon dioxide formed. The factors for calculating these components are obtained as follows:

For tubes *P*:

$$1 \text{ mg. } H_2O \equiv \frac{2 \cdot 016}{18 \cdot 016} \times 11 \cdot 126 \equiv 1 \cdot 2448 \text{ ml. } H_2$$

For tubes *S*:

$$1 \text{ mg. } CO_2 \equiv \frac{28 \cdot 01}{44 \cdot 01} \times 0 \cdot 7997 \equiv 0 \cdot 5090 \text{ ml. CO}$$

For tubes *U*:

$$1 \text{ mg. } H_2O \equiv \frac{16 \cdot 04}{36 \cdot 04} \times 1 \cdot 395 \equiv 0 \cdot 6209 \text{ ml. } CH_4$$

For tubes *V*:

$$1 \text{ mg. } CO_2 \equiv \frac{16 \cdot 04}{44 \cdot 01} \times 1 \cdot 395 \equiv 0 \cdot 5085 \text{ ml. } CH_4$$

If the values for CH_4, as calculated from H_2O and CO_2, are equal, the gas contains no other saturated hydrocarbons. If ethane is also present in the gas both the components may be calculated by the following method from the weights of water and carbon dioxide obtained.

Let the weight of absorbed water be a mg. and of the absorbed carbon dioxide be b mg. If the gas contains X mg. of CH_4 and Y mg. of C_2H_6, the following equations are obtained:

$$\text{For hydrogen content: } \frac{4}{16}X + \frac{6}{30}Y = \frac{2}{18}a$$

$$\text{For carbon content: } \frac{12}{16}X + \frac{24}{30}Y = \frac{12}{44}b$$

from which

$$X = \frac{16a}{9} - \frac{12b}{11} = 1 \cdot 78a - 1 \cdot 09b$$

$$Y = \frac{15b}{11} - \frac{5a}{3} = 1 \cdot 36b - 1 \cdot 67a$$

The volumes in ml. of CH_4 and C_2H_6 are then obtained at N.T.P., using the conversion factors given in the last column of Table I, *i.e.*,

volume of $CH_4 = 1 \cdot 395X = 2 \cdot 48a - 1 \cdot 52b$
volume of $C_2H_6 = 0 \cdot 7371Y = 1 \cdot 035b - 1 \cdot 23a$

The calculation of the results will perhaps be more clearly understood from an actual analysis of a producer gas. In this analysis only one absorption tube was used for each component, except for the absorption of the carbon dioxide corresponding to carbon monoxide, for which two tubes were used.

Volume of gas introduced, v_t	=	100 ml.	
Temperature of water jacket, t	=	24·0° C.	
Barometer reading, b	=	753·0	mm. of Hg
Pressure of gas sample, h	=	+80·0	,, ,,
∴Total pressure of gas sample, $b + h$	=	833·0	,, ,,
Temperature correction for dilatation of mercury (833 mm. of Hg, 25° C.)	=	3·2	,, ,,
∴Corrected total gas pressure	=	829·8	,, ,,
Pressure of saturated water vapour at t° C.	=	22·4	,, ,,
Pressure of dry gas sample at t° C., p	=	807·4	,, ,,

$$\text{Volume of dry gas sample at N.T.P., } v_0 = \frac{807 \cdot 4}{760} \times \frac{273}{273 + t} V_t = 97 \cdot 64 \text{ ml.}$$

The increase in weight of the absorption tubes and the calculated percentages of the gas components are given in Table II.

RESULTS OBTAINED

The accuracy of the method has not been tested by analysis of gases of accurately known compositions. On the other hand, the reproducibility has been shown to be satisfactory by repeated analysis of a single sample of producer gas from a Swedish steelworks. This sample was taken directly from the gas conduit of the producer and was transferred by a pump to a 10-l. pressure container which was filled to a pressure of about 4 atm. The method used for sampling was that given in the A.S.T.M. instructions for taking gas samples.[19]

Table III shows the mean values of 10 gravimetric analyses made on different occasions with this gas sample; results are also given of 10 volumetric analyses of the same gas, carried out in an Orsat apparatus equipped with a compensator tube and with six absorption pipettes and one slow-combustion pipette, using a solution of 250 g. NaCl and 5 ml. H_2SO_4 (sp. gr. 1·50 g./ml.) as the confining liquid.

The values given for the volumetric analyses were obtained by analysing with great care and using every precaution, as prescribed in the instructions for the analysis of producer gas. In ordinary routine analyses the above reproducibility is not generally attained.

DISCUSSION

The gravimetric gas-analysis method described was specially developed as a control method in a research

Table III

RESULTS OF ANALYSES OF PRODUCER GAS

All values in vol.-% of dry gas

	CO_2	O_2	C_nH_m	H_2	CO	CH_4		
						Calc. From H_2O	Calc. From CO_2	Mean Value
				Gravimetric Method				
Mean value of 10 analyses	3·46	0·10	0·21	15·73	29·46	2·44	2·53	2·49
Highest value	3·53	0·22	0·25	15·95	29·81	3·28	3·16	3·22
Lowest value	3·34	0·00	0·16	15·21	29·13	1·75	1·69	1·72
				Volumetric Method				
Mean value of 10 analyses	3·45	0·10	0·20	15·58	29·56		2·80	
Highest value	3·67	0·25	0·50	15·77	30·20		3·00	
Lowest value	3·23	0·00	0·00	15·27	28·83		2·51	

on gas analysis started by Jernkontoret in 1950 to improve the procedure for the volumetric analysis of technical gases. The method proved to be entirely satisfactory for this purpose.

On comparison of the results obtained by the gravimetric and the volumetric method, the advantages of the former may be doubted. The two methods have apparently given the same degree of accuracy, and the gravimetric method seems at first sight to be more complicated. However, this is not likely to be the case. Thus, before a run of analyses with an Orsat apparatus, equilibrium must be established in all absorption pipettes between the gas components physically dissolved in the liquid absorbents and the gases in the pipettes; this is usually done by carrying out two or three analyses of a gas of a similar type as the gas samples to be analysed.

In the gravimetric analysis a similar procedure for reaching equilibrium conditions is unnecessary, because gas components possibly dissolved in the absorbents are removed by flushing with nitrogen. Different types of gas can therefore be analysed in immediate succession, and this is a considerable advantage in analysing single samples of unknown composition. In addition, some errors necessarily entailed in the volumetric methods are avoided by the gravimetric method.

The gravimetric apparatus is also less difficult to handle than the volumetric one. In the gravimetric method the troublesome forcing of the gas back and forth into the absorption pipettes by raising and lowering the levelling bulb is eliminated, and the manual work, except for the introduction of the sample into the apparatus, is limited to the connection, disconnection, and weighing of the absorption tubes, the net time for the analysis work thus being considerably reduced.

An account of a gravimetric method of gas analysis recently published by Barthauer, Haggerty, and Friedrich[20] came to the notice of the author after the main work on this paper was finished. That method, which was developed for the analysis of 'synthesis gas' consisting of a mixture of CO_2, CO, H_2, and CH_4, is also based on the combustion of sample components and absorption of combustion products, the combustion being effected by a catalyst of platinum–palladium dispersed on silica gel. The method is said to be "rapid and very accurate" and decidedly superior to both Orsat and mass-spectrographic techniques.

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The Universal Decimal Classification Applied to Metallurgical Literature

By Einar Öhman and J. P. Saville

SYNOPSIS

The authors explain the need for a rapid and accurate means of classifying metallurgical literature and, after discussing other systems, describe the Universal Decimal Classification, its construction and present state, and the means by which it is revised and kept up to date. The contributions made by The Iron and Steel Institute and Jernkontoret in revising the metallurgical sections are explained. The article concludes with a description of a method for making the system better known and more useful to metallurgical workers, by producing "Selected Schedules from the U.D.C." for their special guidance. 934

UNTIL fairly recent times, the word 'metallurgy' has been understood to mean, in general, the extraction of metals from their ores; the "Iron and Steel" volume of Percy's celebrated "Metallurgy" deals almost entirely with the production of the metals, and devotes relatively little space to manufacture and heat-treatment. The word is still used in this earlier sense in the title of the *Institution of Mining and Metallurgy*.

But with the growth of chemical knowledge in the early nineteenth century, the reactions involved in iron- and steel-making and the production of other metals began to be better and more widely understood; when Sorby applied his petrographic-microscope technique to the study of iron and steel it became possible to form a picture of the interior of metals, and 'metallurgy' as we know it today began to develop.

The Necessity for Classification

Metallurgy is now founded upon a number of exact sciences, the basic ones being concerned with the chemical behaviour and the internal structure of metals; it has become of great social importance, involving not only chemists, physicists, and engineers, but also manufacturers, merchants, economists, teachers, and historians. Its study includes mechanical working, corrosion studies, electrical and mechanical engineering, geology, chemistry, physics, metallography, photography, economics, statistics—and more besides.

Though each of these aspects may be the life-time specialization of an individual, it is probable that most metallurgical workers will sooner or later find themselves concerned with more than one, and will want to know where to turn for information on each. The sources of information may be extremely varied; at the beginning of a study a standard text-book, which gives the accepted theories or describes the usual methods of working, may be sufficient, but in many fields progress has left the text-book behind and, to keep up with recent knowledge, it is necessary to read the current periodical literature or to study reports of research. This has never been a light task and could become a full-time one, to the exclusion of any genuine scientific work, were it not for the growth of information departments, the establishment of abstracting services which cover various fields of knowledge, and the regular publication of these abstracts—either as part of a periodical, as in the case of the *Journal of The Iron and Steel Institute*, and *Stahl und Eisen*, or separately, like the *Bulletin Analytique de la Centre de Documentation Sidérurgique*, *Metals Review*, and *Jernkontorets Litteraturöversikt*.

It is well known that there are two main forms of abstract: the *indicative* and the *informative*. Indicative abstracts tell the reader that the paper is one that he should read, whilst informative abstracts provide a summary which may often render it unnecessary to read the whole paper. Each form has its advantages and there can be no fixed rule about which treatment should be applied; many, but by no means all, of the abstracts published in the *Journal of The Iron and Steel Institute*, for example, are indicative, whereas many of those published in *Jernkontorets Litteraturöversikt* are informative. There is no doubt, however, that abstracts in general are extremely useful: they satisfy the immediate need of the reader by

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informing him of results or by telling him where information may be found. In addition, they are of great permanent value.

Research workers frequently require a 'bibliography,' *i.e.*, a list of abstracts, or of references alone, dealing with a given subject, and expect this to be compiled by the information department of their firm or research institute or by that of the professional institute of which they are members. A request, which is the opposite of this comprehensive survey and is made almost as frequently, is for the one paper —by a particular author, issued in a certain year, or published in a given journal—which contains unique and necessary information. Both types of request can only be answered by the use of some system which makes it possible to refer to all the material available to the information department. There is no great difficulty with text-books, as their titles are usually a sufficient guide to their contents, but the rapid growth of periodical literature and the immense quantity of specialized papers now being published, make the classification of abstracts a more difficult problem.

Special Classification Systems for Metallurgical Literature

It is therefore natural enough that classification systems should have been invented which do not attempt to cover the whole of human knowledge, but deal with very special fields and subdivide them in great detail. The International Institute of Welding, for example, uses in its *Bibliographical Bulletin for Welding* a special classification covering the technique, apparatus, and auxiliaries for welding, and including a large number of other fields, treated largely as applications of welding.

In the metallurgical field two such special systems have been published recently, the French *Classification Alpha-Numérique*[1] and the American *ASM/SLA Metallurgical Literature Classification*.[2] The French system was devised by Monsieur H. Malcor and is used by the Centre de Documentation Sidérurgique in Paris for their *Bulletin Analytique* and for their central card-index of abstracts. The American system was worked out by a joint committee of the American Society for Metals and the Special Libraries Association ; it has been used since 1950 by *Metals Review* for the classification of its abstracts.[3] The ASM/SLA system is intended primarily for use with marginally-punched cards and mechanical sorting, though it can be equally well used with other filing systems ; the idea of mechanical sorting has, however, very strongly influenced the grouping of subjects. There are three main groups : the *Processes and Properties Index*, the *Materials Index*, and the *Common Variables Index*. An example may be quoted from each index : from *Processes and Properties* F.23.q means 'finish rolling,' from *Materials* Al.j.45 is equivalent to 'aluminium alloy 75 S,' and the *Common Variable* 4.3 indicates 'sheet, strip, and plate.' Now by separating out all those cards which are punched on the top edge for F.23.q, on the left-hand edge for Al.j.45, and on the right-hand edge for 4.3, we obtain all the cards in the file which relate to 'the finish rolling of 75 S sheet.' The same result could, of course, be obtained if the cards were filed in alphabetical and numerical order under some such complex notation as F.23.q. : A.j.45 : 4.3.

This system is excellent for anyone who is only concerned with information on materials and their production, fabrication, and uses ; but an important specialist like the iron- and steel-works' engineer will find that suitable subdivisions for various types of machinery—rolling-mills, presses, hammers, manipulators, and so on—are lacking. There is only one heading in the *Common Variables Index* that would suit him—1.2, 'production machinery'; by combining this with the symbol for 'rolling' in the *Processes and Properties Index*, he can obtain 'rolling-mills' but it is not possible to subdivide this, even into 'two-high' and 'three-high.' In common with other specialists he will, in fact, have to prepare his own 'common variables.' This adaptation of the system to individual requirements seems to be intentional, for the detailed breakdown of the *Processes and Properties Index* is also left to the discretion of the specialist. While this breakdown may assist the specialist, anyone dealing with material of very varied nature will be more interested in a comprehensive system which, even with some sacrifice in detail, may be used for all the information to be classified.

It has been suggested that the ASM/SLA system should be more widely adopted by metallurgical information centres and abstract publications, but this is scarcely possible, since many of them are not only concerned with metallurgy in its specific sense, but with literature in adjacent fields. Some have an equal interest in mining, others devote attention to fuels or to rolling-mill design, others again to physical metallurgy or to the applications of metallic materials. Frequently, the economic, trade, and social problems of the metallurgical industries are dealt with ; the following selection of headings from one issue of the weekly news-letter of a large Swedish concern, which specializes in steel and in wood-products, is illuminating :

Amortization	Responsibility of Post Office
Export credit	(registered letters)
Real-estate taxation	Plan for highway development
Investment funds	Declaration of income in 1954
Farm taxation	Sickness insurance
Customs tariff	English-Swedish trade agree-
Trade statistics	ment, 1954
Time study	Safety clothing
Production trends	Industrial relations
Industrial sanitation	Labour agreement of Swedish
Statistical analysis	machine industry.

More technical headings are :

Ceramic materials	Maintenance of transmission
Bearing tolerances	lines
Grinding of carbide	Transformer oil
tools	Mine locomotives
Chromatography	Microspectroscopy
Plasticizers	Patternmaking
Water pollution	Hardness testing of thin sheet
Fluorescent lamps	Controlled atmospheres
Induction heating	Wire drawing.

No special classification could deal with such a range of subjects which, chosen at random, give only an incomplete picture of the variety of information that special librarians in the metallurgical and allied

industries must distribute and must also classify and file for future reference.*

It is the authors' firm belief that a *universal* classification system is the only possible solution, and that among such systems the Universal Decimal Classification (U.D.C.) is the only one that can be seriously considered.

This system is widely used in a number of countries and is adequately supported by an international organization for its continuous revision and development ; it has been found possible to introduce improvements of a fundamental character into the U.D.C., and it is probable that the valuable ideas of Bliss,[4] Ranganathan,[5] and others, may be made use of in this way.

The History of the Universal Decimal Classification

The idea of using a decimal scale to indicate the subdivisions of a classification system originated with Melvil Dewey, who was then sub-librarian of Amherst College, Massachusetts ; he developed his system in 1873, and it was first published in 1876. The ' Dewey Classification ' has been progressively developed since then, and is now used by more than 90% of the libraries in the United States, and by many in the United Kingdom.

On the initiative of two enthusiastic Belgians, P. Otlet and H. La Fontaine, a scheme was launched in Brussels in 1895 for establishing a comprehensive classified index to published information. A classification system was needed for this index and, upon the foundation of the International Institute for Bibliography (I.I.B.), it was decided to use the Dewey system as a basis. The I.I.B. rapidly expanded the schedules, by arrangement with Mr. Dewey, and published the first international edition of the Universal Decimal Classification in 1905 : it contained 33,000 subdivisions. The classification has grown steadily since then ; the First and Second World Wars interrupted its development, but between the wars international collaboration was restored. It is once again on a firm footing.

The second international edition, printed as the first in the French language under the editorship of MM. Otlet and La Fontaine, was published between 1927 and 1933 and contained some 70,000 subdivisions. The sections on Pure and Applied Science, with which metallurgists have the most to do, were edited by Ir. F. Donker Duyvis, who is now the Secretary-General of the Fédération Internationale de Documentation (F.I.D.), the name by which the original I.I.B. is now known. Later editions are the third, in German, begun in 1933, the fourth, in English, begun in 1936, and the fifth, once again in French, begun in 1939. The German edition is now complete ; the last volume of the alphabetical index appeared in 1953. The others, having been interrupted by the War, are not yet complete ; to some extent they supplement each other and there is some overlapping. Where there are schedules in more than one language, however, international collaboration has ensured that there are no differences of meaning.

In addition to the texts of the full schedules, there are abridged editions in all the major European languages. These form valuable introductions to the use of the classification ; as will be shown later, abridgment by the shortening of numbers does not lead to the omission of concepts, and the abridged versions are therefore still very useful. In addition, the Introduction to the Abridged English Edition (British Standard 1000A : 1948) contains a brief history of the U.D.C. and excellent notes on its arrangement.*

The Scope and Construction of the U.D.C.

There are three main principles in the U.D.C. The first is classification by the analysis of ideas ; by this, related concepts and groups of concepts are brought together, and the arbitrary and haphazard systematization of many other methods is avoided. The second principle is universality, in that an attempt is made to include every subject of human knowledge, and to group these subjects from every point of view. No narrow ' specialist ' fields are developed ; this principle is in complete opposition to the notion that a specific specialized subject may be isolated and set aside from all other specialized fields. The U.D.C. permits the relation of any special subject to any other branch of human thought, and allows for the classification of individual ideas from different viewpoints. This is inherent in the system of analytical numbers having special symbols which denote different commonly-occurring viewpoints, and the simple means by which numbers indicating single ideas may be linked to make more complex ones.

The third major principle is the decimal one. The whole of human knowledge is divided into ten groups, and each group is subdivided by the addition of decimal places ; the use of Arabic numerals for symbols readily permits this, and makes the introduction of new subdivisions very simple. It also facilitates, to some extent, learning by heart. It does not follow, however, that all the systematic division of a subject will occur in tens ; such a mechanical approach is foreign to the U.D.C. Where fewer subdivisions will do, no attempt is made to stretch the process to a greater extent ; the subject itself decides the openness of its scale.†

The primary divisions of the U.D.C. are still as Melvil Dewey laid them down. Group 0 is *Generalities*, relating to documentation and bibliography : for example, 016 is Specialist Bibliographies and 025.45 the U.D.C. itself. Group 1 is *Philosophy*, the starting-point of critical speculative thought. Group 2 is

*The ASM/SLA system is employed by the British Iron and Steel Research Association (*see* P. E. Colinese : *Aslib Proceedings*, 1953, vol. 5, pp. 345–348) but it is only applied to the Association's own reports and is intended purely for internal use ; the amount of material is small and not likely to increase very much.

*A more detailed account of the U.D.C. is given in S. C. Bradford : " Documentation " : 1953, 2nd edition, London, Crosby, Lockwood and Son Ltd.

†Further details of the general principles of the U.D.C. are given in : " Guiding Principles, Rules and Procedure for the Development and Revision of Documentary Classification, with Special Reference to the Universal Decimal Classification (U.D.C.) " : 1953, The Hague, Fédération Internationale de Documentation.

Religion, since the individual mind considers first the all-embracing, before examining the multiplicity of phenomena. Group 3, the *Social Sciences*, deals with human relations, and includes Political Science (32), Law (34), Education (37), Trade and Communications (38). Group 4 is *Language* or philology, the science of verbal communication, the prelude to Group 5, *Pure Science*, and Group 6, *Applied Science*. Pure Science begins with Mathematics and goes on through Physics (53), Chemistry (54), and Geology (55), to conclude with Zoology. The first of the applied sciences is applied biology, that is, medicine, but the engineering sciences are dealt with next (62). These require much subdivision : 621, Mechanical Engineering, includes Steam and Steam Engines (621.1), Water Power (621.2), Electrical Engineering (621.3), and Internal Combustion Engines (621.4), before Workshop Practice (*i.e.*, most working and fabricating processes) is reached at 621.7 to 621.9 ; Mining is 622, Civil Engineering 624, Land Transport 625, Rivers, Ports, and Roadsteads 627. Agriculture is 63, Commerce and Communications 65 ; Chemical Technology (66) includes Metallurgy in the older sense (669) ; Manufactures (67), Mechanical Trades (68), and the Building Industry (69) close this Group. Group 7 includes, as well as Architecture (72), Sculpture (73), and Drawing and Industrial Art (74), Photography (77) both artistic and technical (high-speed cinematography, for example, is 778.534.8). The main groups finish with Group 8, *Literature*, and Group 9, where are to be found *Geography*, *Biography*, and *History*. The order of these main groups is not of great importance, but it will be seen that Dewey imposed a reasonable logic upon it.

At the end of the main tables come the Auxiliary Numbers and Symbols ; by these relatively simple means the U.D.C. has been made universal, in the sense that it allows of almost infinite combination and modification of concepts. The most important of these devices is the colon (:), known as the ' sign of relation,' which enables complex ideas, such as ' the chemical analysis of open-hearth slags,' to be constructed by linking the numbers for ' chemical analysis ' and ' open-hearth slags.'

Numbers combined with certain symbols are used to denote general aspects, for example :

Language	=20	Publication in English
Form	(045)	Article in a periodical
Place	(485)	Events or practice in Sweden
Time	" 19 "	20th century
Viewpoint	.002.6	Products of a process
	.003.2	Financial aspect.

In addition to these there are special subdivisions of the form .0*xx* (where *x* may be any number), confined to smaller sections of the classification and representing concepts common to restricted ranges of subjects. Finally, there are 'hyphen-auxiliaries' denoting subsidiary concepts applicable in certain broad technical fields ; for example, parts common to many types of machine, or shapes in which many kinds of material may be produced.

At this point, a few examples will be given to illustrate subdivision :

Example 1

5	Pure Science
53	Physics
539	Molecular and Atomic Physics
539.4	Resistance to Stress
539.41	Resistance to Elastic Deformation
539.414	Resistance to Torsion.

Example 2

6	Applied Science
62	Engineering Science
621	Mechanical Engineering
621.7	Workshop Practice
621.74	Foundry Practice
621.745	Foundry Melting Practice.

Example 3

6	Applied Science
66	Chemical Technology
669	Metallurgy
669.1	Ferrous Products in General
669.16	Production of Pig-iron and Ferro-alloys
669.162	Production of Pig-iron
669.162.2	The Blast-furnace
669.162.26	Operation of Blast-furnaces
669.162.266	Tapping
669.162.266.2	Tapping of Pig-iron
669.162.266.23	Tapping into Pig-beds
669.162.266.235	Pig-breaking. Pig-breakers.

In these examples each extra digit adds a further element of precision ; it will also be seen that the shorter numbers include the subjects of the longer ones, so that the degree of precision may be adjusted to the needs of the user. A few further examples will show the value of the colon (:) method of connection, of the hyphen-numbers, and of some of the auxiliary symbols :

Example 4

(*a*) 621.785 Heat-treatment ; (*b*) 669.14.018.8 Stainless Steel

(*c*) 621.785 : 669.14.018.8 The Heat-treatment of Stainless Steel.

Example 5

(*a*) 669.131.6 Grey Cast-iron ; (*b*) –462 of Tubular Form

(*c*) 669.131.6–462 Grey Cast-iron Pipe.

Example 6

(*a*) 669.183.2 The Open-hearth Process ; (*b*) .003 Economic Aspects

(*c*) 669.183.2.003 Economic Aspects of the Open-hearth Process.

Example 7

(*a*) 669.162.4 Electric Pig-iron Furnaces ; (*b*) .041.532 Low-shaft Furnaces (an auxiliary number from the series common to 66, Chemical Technology) ; (*c*) (485) Sweden

(*d*) 669.162.4.041.532(485) Swedish Low-shaft Electric Pig-iron Furnaces.

Example 8

(*a*) 669.15–194 Low-carbon Ferrous Alloys ; (*b*) 669.24 Nickel ; (*c*) 669.26 Chromium

(*d*) 669.15.24.26–194 Nickel-chromium Alloy Steel.

Example 4 shows the special value of the colon system for cross-referencing ; group (*c*) will be filed with all other references to Heat-treatment and when it is transposed and written as 669.14.018.8 : 621.785, it will be filed with all other references to Stainless Steel. It can thus be readily picked out, from whichever angle it is approached. In each of Examples 5 to 8, the original concepts (*a*) are made more precise by auxiliaries.

The Future Development of the U.D.C.

Earlier paragraphs have emphasized the extensive development of the U.D.C. in the last fifty years ; it should not be assumed, however, that development has ceased. The U.D.C. is intended to be as wide and as deep as human knowledge is or may become ; it must therefore expand as human knowledge expands and be revised as knowledge is revised, so that its concepts may not be based on theories which have been abandoned, or on processes which are obsolete. This revision and extension involves continual supervision, and complete agreement among the supervisors ; in practice the supervisors are found among the users of the U.D.C. in many countries. They put forward their suggestions to the F.I.D., through their national organizations, and, in its turn, the F.I.D. circulates all member countries. This system gives a reasonable period for objections, so that no fresh schedules are introduced, nor old ones cancelled, without consideration by expert users. Nor—and this is an essential feature—are existing numbers given new meanings in the course of revision; *extensions* of meaning are often desirable but, while a number may be cancelled and its concept given a new number, the old number will not be used again in a new sense for ten years.

However, even within this scheme of co-operative control conflicting schools of thought have emerged. Some users resist any kind of change in the U.D.C., except for minor additions ; they strongly oppose any large-scale rearrangement. Others tend to the opposite view in an attempt to allow for recent scientific or technical development, but often go too far in a desire to rearrange the schedules in the interests of logic alone. It is sometimes forgotten that the logic of a schedule does not appear quite the same to each individual user, that the logical order of concepts is often less important than that there should be a place for each of them, and that any rearrangement of the shorter, and therefore more comprehensive, numbers will alienate the wide circle of general users as well as other specialists.

However, the majority of active U.D.C. users and revisers have approved a compromise between these two tendencies : they endeavour to make the classification better for present users and more attractive to non-users ; to reduce confusions in the existing tables, and to introduce new concepts without disturbing the tables too much. This may slow down the process of revision but it produces the least dissatisfaction.

The Rôle of The Iron and Steel Institute in Revision

The Iron and Steel Institute has taken part in all the post-war developments of the parts of the U.D.C. which deal with metallurgy and metal-working : this was the natural outcome of the decision to use the system in the Institute's Information Department. The Institute is represented on the British Standards Institution's Sub-committee OC/20/4—Universal Decimal Classification, and on the appropriate U.D.C. panels of the Sub-committee. In Great Britain the B.S.I. is the responsible body for U.D.C. matters and is editing and publishing the fourth international edition (English) of the classification. For all purposes other than U.D.C., Aslib (the Association of Special Libraries and Information Bureaux) is the national member-body of the F.I.D.

The Institute has, therefore, very close links with the centre of U.D.C. activity. The section 669, Metallurgy, was brought to a satisfactory state by 1948, but the sections 621.7 to 621.9, which deal with the working of metals, proved rather more refractory, because of strong pressure, both from those who resist change and those who advocate radical change ; this made progress in any direction extremely difficult, but, after some years' struggle, a satisfactory solution has now been reached. A British draft was presented and accepted at the 1952 Congress of the F.I.D. in Copenhagen ; the schedules had been partially filled in by the time of the 1953 Congress in Vienna where it was decided that, in general, they should be completed on the British lines.

Final agreement was reached at a meeting held at the British Standards Institution in December 1953, where The Iron and Steel Institute, Jernkontoret, the Verein Deutscher Eisenhüttenleute, the Deutscher Normenausschuss, and the B.S.I. were all represented. In earlier stages of the discussions, representatives of users of the U.D.C. in France, Holland, Austria, and Switzerland had taken part ; this does not exhaust the list of bodies with whom contact has been made over U.D.C. matters at one time or another, but it does indicate that liaison is particularly strong with the major European metal-producing countries.

The Rôle of Jernkontoret in Revision

The Swedish Society for Technical Documentation (Tekniska Litteratursällskapet or T.L.S.) is the Swedish national member of the F.I.D. and all U.D.C. work in Sweden is centred in its U.D.C. Committee. In 1948 it was decided to adopt the U.D.C. for the classification of the abstracts published in *Jernkontorets Litteraturöversikt*, the abstract journal of Jernkontoret. Since that time, members of the staff of Jernkontoret have been actively engaged in the work of the T.L.S., in the U.D.C. and in other fields, and since 1952 one of the authors (E.Ö.) has been president of the Society.

A few years ago, the T.L.S. began an arrangement whereby the abstracts of Jernkontoret, and those of other Swedish abstracting bodies, are made available in card form ; these, and the synopses of technical papers published in some 40 Swedish periodicals, are reproduced by a near-print method. By this means, Swedish industries can build up a card index, uniformly classified according to U.D.C., and containing a very complete bibliography of Swedish technical literature, as well as some selected abstracts, within

certain fields of activity, of articles published in other countries. This has been made possible by the general adoption of the U.D.C. by the Swedish technical press and special libraries, and has led to the establishment of fairly uniform and consistent classification practice.

Extensive contributions have been made from the Swedish side to the general revision and development work which has been going on in the U.D.C. in the last few years, and representatives of Jernkontoret have been very active in this. The section on Mining (622) in the fourth international edition (English) of the U.D.C. has just been published ; the draft of this was studied in detail at Jernkontoret, and numbers of suggestions were made which were incorporated in the final version. One of the authors (E.Ö.) submitted a paper on the classification of materials to the 1951 F.I.D. Congress in Rome, and it was also discussed at Copenhagen in 1952, where it met with general approval. He also worked out, with the aid of other members of the staff of Jernkontoret, extensive proposals for the revision of certain sections of Metallurgy (669), Metallography (620.18), and Powder Metallurgy ; after thorough revision by the present authors and Herr H. Dickmann of V.D.Eh., the proposals were presented to the 1953 F.I.D. Congress in Vienna and finally edited at the London Meeting mentioned above. The proposal for Powder Metallurgy has been incorporated in the British draft of 621.7/.9, and the others have been submitted to the F.I.D. as proposals for extensions.

Presentation of the U.D.C. to Metallurgists at Large

It is hoped that the description of the U.D.C. given in this paper has removed any misconceptions about its suitability for the classification of metallurgical literature, and that it has been made clear that these arise, not from any weakness in the system itself, but from the quite recent and enormous expansion of meaning that the word ' metallurgy ' has received, and the wide range of information needed by metallurgical industries. It is not surprising that someone from the metallurgical industries should expect to find all the information of interest to him under the heading Metallurgy ; but a little study of the U.D.C. will soon show him what he may expect to find there, and in which sections he must look for other special information. In its classical sense, the concept ' metallurgy ' is to be found in 669 ; the primary production of iron, steel, and other metals and alloys is worked out there in considerable chemical and technical detail. Also in this section are arranged the classification of alloys according to composition and to use, and the thermal transformation products of the alloys of iron with carbon and other metals. However, to find the properties of metals and alloys one must refer to Physics (53), and for the determination of properties to Mechanical Testing (620.1), because these concepts are not confined to metals and alloys, but are common to all materials. Similarly, though they may be essential to the producers of sheets and sections, one must not expect to find ' rolling-mill motors ' in Metallurgy ; Electrical Engineering has its proper place (621.3) as a science in its own right.

It seems, therefore, that a *guide* to the U.D.C. would be very useful : this guide would take into account all the possible fields of interest of all types of metallurgists, and of non-technical members of the staffs of industrial firms, and lead them to the appropriate places in the schedules. With this idea in mind, one of the authors (J.P.S.) has made a preliminary draft entitled " Proposed Schedules for the Classification of Metallurgical Literature by means of the Universal Decimal Classification." Parts of this have already been discussed with the co-author and Herr H. Dickmann of V. D. Eh. When completed, it will contain the complete schedules on the mining, extraction, and working of metals, connected by abridged schedules on other necessary subjects such as physics, chemistry, geology, electrical engineering, the social sciences, and so on ; in addition, tables of the auxiliary numbers and symbols, and full instructions for use will be given. It will be produced in English, Swedish, and German, and possibly in French and Spanish also. The texts will either be bound together as a single inclusive volume or appear as separate parts of a single work. The precise layout and format are not yet decided.*

It is hoped this publication will make the classification of metallurgical literature far easier and, what is after all the aim of classification, reference to it speedier and more accurate. The U.D.C. is not the only possible classification for metallurgical literature, or for the literature of any other subject, general or specialized, but it has the very great advantage of construction, supervision, and revision by people who are expert in the subjects themselves as well as in the U.D.C. itself. Its international control and development are not its least merits ; the classification is already widely used in the metal-producing countries of Europe, and wider use must be of service to its present users, to those who adopt it in the future, and in the international exchange of scientific and technical knowledge.

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*A similar publication for the building industry, recently published under the auspices of the International Council for Building (Conseil International de la Bâtiment) is : " A.B.C. Abridged Building Classification for Architects, Builders, Civil Engineers ; a Selection from the Universal Decimal Classification ." : 1953, Tumba, Sweden, the Council.

SYNOPSES OF PUBLISHED SWEDISH PAPERS

In addition to the papers published in this issue of the *Journal*, a number of other papers were specially prepared in connection with the meeting in Sweden. The synopses of these papers, which were published in the October and November, 1953, and April, 1954, issues of the *Journal*, are repeated below for reference.

SINTERING PRACTICE AT DOMNARFVET, SWEDEN

By Christer Danielsson, Met.E.

The sinter plant and practice at the integrated iron and steel plant at Domnarfvet, Sweden, are outlined, and the relevant details of blast-furnace practice are also included. The resulting iron production rate is stated to compare favourably with that of good American furnaces. (*October*, 1953)

THE MECHANISM OF REDUCTION OF IRON OXIDES

By J. O. Edström

The reduction of pure, natural hematite and magnetite crystals by hydrogen and carbon monoxide was studied mainly with the aid of the microscope. The more rapid reduction of Fe_2O_3 is explained by an earlier and more extensive formation of pores in the reaction products. The low strength of pure hematite ores and of well-oxidized, burnt, rich-ore pellets during reduction with carbon monoxide is due to the formation of pores, accompanied by a considerable expansion and a disintegration of the structure.

The minimum reaction velocity above 600° C. when Fe_2O_3 and Fe_3O_4 are reduced by hydrogen is associated with the appearance of dense continuous layers of wüstite.

The mechanism of reduction and the cases of the different pore-formation tendencies are discussed. (*November*, 1953)

STRUCTURAL CHANGES DURING ANNEALING OF WHITE CAST IRONS OF HIGH S: Mn RATIOS, INCLUDING THE FORMATION OF SPHERULITIC AND NON-SPHERULITIC GRAPHITE AND CHANGES IN SULPHIDE INCLUSIONS

By Axel Hultgren and Gustaf Östberg

The mechanism of formation of dispersed graphite flake nests, compact graphite nests, and graphite spherulites during annealing at 900–1150° C. of white cast irons of different, mainly high, S : Mn ratios was studied by examining the microstructures of quenched specimens.

Any one of the three types of graphite aggregate mentioned could be produced at will by a suitable combination of S : Mn ratio of the iron, hydrogen content of the annealing atmosphere, and annealing temperature.

Several interface combinations, not only those involving sulphide, appeared to favour nucleation of graphite, with basal-plane–interface coincidence, and growth with such coincidence maintained locally. In this way, an iron or manganese sulphide inclusion could serve as a nucleating particle for a graphite spherulite provided that the radial texture established by oriented nucleation and growth was preserved during further radial growth.

The evidence indicates that for this purpose an agency is probably required which would sufficiently retard the *a*-axes growth in relation to the *c*-axis growth of the graphite. Sulphur and/or hydrogen appear to exert such an effect when present in sufficient amount.

Changes in constitution and shape of sulphide inclusions during annealing have been studied, and also the changes in shape during growth of adjoining graphite. Under certain conditions of composition and heat-treatment, manganese sulphide undergoes a diffusionless transformation into an anisotropic form during quenching from a high temperature. (*April*, 1954)

EUTECTIC SOLIDIFICATION IN GREY, WHITE, AND MOTTLED HYPO-EUTECTIC CAST IRONS

By A. Hultgren, Y. Lindblom, and E. Rudberg

Solidification under controlled conditions was studied in four hypo-eutectic cast irons, conditions being selected to produce coarse graphite, fine ' undercooled ' graphite, a mottled structure, and a white iron. Samples were quenched at predetermined points on the cooling curves, and sections were examined for evidence of the processes occurring during cooling. With small samples and drastic quenching, regions that had not been reached by reaction formed a microstructure of fine ' quench ' ledeburite, which contrasted with any ' furnace ' structure formed during furnace cooling. The graphite–austenite constituent, of eutectic composition, of such furnace structures generally had the form of rounded rosettes ; its formation was associated with a characteristic cooling curve, and its fineness was closely correlated with the temperature of its formation. The formation of cementite–austenite eutectic was associated with a cooling curve of different shape. In the mottled iron investigated, graphite–austenite was formed in specimens quenched in the early stages of cooling, corresponding to a 'graphitic' cooling curve. In the later stages of cooling, the curve assumed the form characteristic of cementite–austenite formation ; this was also revealed by the microstructure of specimens quenched at this stage.

In the irons studied, the appearance of the graphite–austenite aggregates, their composition, and their general manner of development, related to the cooling curves, indicate direct formation from the melt by eutectic reaction. (*April*, 1954)

METALLOGRAPHY OF DELTA-FERRITE. Part I—EUTECTOID DECOMPOSITION OF DELTA-FERRITE

By Kehsin Kuo

The δ-ferrite in a high-molybdenum steel that has been heated to 1200–1300° C. was found to decompose in a eutectoid manner within the temperature range 900–1150° C., giving austenite and the Fe_3Mo_3C carbide which is here called δ-eutectoid. The morphology and kinetics of the formation of this eutectoid have been

studied by continuous and isothermal transformations, and the formation has been found to be a nucleation-and-growth process with the Fe_3Mo_3C carbide as the nucleating phase. The transformation diagram has been plotted, and displays the common *C* shape of the pearlite reaction. Special attention has been paid to the parallelism between the formation of this eutectoid and that of pearlite.

Besides this eutectoid decomposition of δ-ferrite, two closely related reactions also occur. As more austenite is formed from δ-ferrite, the austenite retained from high temperatures becomes unstable and later transforms allotropically into ferrite. At lower transformation temperatures a general precipitation of Mo_2C occurs in δ-ferrite, overlapping the δ-eutectoid transformation. These two reactions also have the *C*-type transformation diagram.

It is suggested that the slow diffusion of molybdenum is the controlling factor for these three internally connected transformations. It was expected that any alloy element which forms a special carbide and at the same time promotes the formation of δ-ferrite—as do Cr, W, and V—would make the formation of this δ-eutectoid possible, and this idea was confirmed experimentally. (*April*, 1954)

THE GROWTH OF NODULAR GRAPHITE

By M. Hillert and Y. Lindblom

It is suggested that graphite spherulites grow by screw dislocations generated by the inclusion of foreign atoms in the graphite lattice. In support of this theory rare-earth elements are shown, by means of autoradiography, to be uniformly distributed through the spherulites present in a nickel–carbon alloy treated with *mischmetall*. (*April*, 1954)

PRACTICAL EXPERIENCE IN THE USE OF REPEATERS IN A LOOPING MILL

By Hans Wilhelm Riddervold

The paper gives an account of the factors which affect the working reliability when rolling with repeaters in 3-high and alternating 2-high mills, and deals in detail with the sizes of the reductions, the guides, and the repeaters. (*April*, 1954)

THE NEW FAGERSTA WIRE-ROD MILL

By K.-E. Pihlblad

The paper describes the layout and operation of the Fagersta wire-rod mill and a detailed account is given of the methods used to achieve exact roll setting for the Leufvén mill stands. (*April*, 1954)

ROLLER-BEARINGS IN SWEDISH ROLLING MILLS AND THE S.K.F. ROLLING-MILL DESIGN

By Axel Leufvén

A short outline of the development of the roller-bearing designs in Swedish rolling mills is given. The importance of the S.K.F. oil-injection method is emphasized, and an account is given of modern bearing applications in different types of rolling mill. The origin and development of the S.K.F. rolling-mill design with preloaded bearing housings is described, as well as the use of this design for wire-rod and cold-rolling mills. (*April*, 1954)

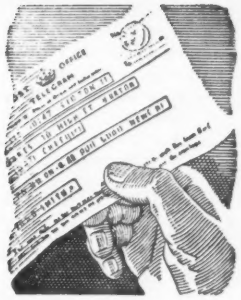
NEW SWEDISH MILL DESIGNS AND LAYOUTS FOR MEDIUM AND SMALL SECTIONS AND WIRE ROD

By S. Erik M. Norlindh

The development of rolling-mill technique in Sweden has been mostly directed towards the need of the small high-grade steelworks for modern but nevertheless economical rolling mills for bars and wire rods. This has been possible owing to the development of repeater rolling to a high degree of reliability, even for breakdown ovals and leader ovals down to 0·2-in. wire rod at final speeds of up to about 4000 ft./min.

A new layout for a combined rolling mill for medium and small sections and wire rods is described, in which the most recent phase of development is the ability to roll small ingots. An account is given of the essential new designs that have made this development possible. (*April*, 1954)

N E W S



ANNOUNCEMENTS AND NEWS OF SCIENCE AND INDUSTRY

PROGRAMME FOR SPECIAL MEETING IN SWEDEN, 1954

The Council have accepted an invitation from the Council of Jernkontoret, Stockholm, to hold a Special Meeting of The Iron and Steel Institute in Sweden from **Monday, 7th June, to Friday, 18th June, 1954.**

For the convenience of those members who do not wish to attend over the whole period, the meeting has been divided into three sections.

The programme is as follows:

SECTION I—GOTHENBURG AND VÄSTERÅS

Monday, 7th June (Gothenburg)

Morning: Members and Ladies arrive by steamship at Gothenburg.

Mid-day: *Excursion* 1 (Members and Ladies)—Visit to Långedrag, with lunch by invitation of the Swedish Lloyd Steamship Co., Ltd. A short sightseeing tour by bus will be made on the return journey to Gothenburg. (*Note:* Only Members and Ladies who have travelled by Swedish Lloyd from Tilbury to Gothenburg are invited to take part in this excursion.)

Evening: Free in Gothenburg.

Tuesday, 8th June (Gothenburg)

Members and Ladies may take part in *either*:

All day: *Excursion* 2—AB Ferrolegeringar, Trollhättan, with lunch and sightseeing, including visit to hydro-electric power plant.

or

Morning: Members may take part in *one* of the following excursions with lunch:

Excursion 3—AB Svenska Kullagerfabriken (S.K.F.).
Excursion 4—AB Volvo.
Excursion 5—AB Götaverken.

Ladies: *Excursion* 6—Visits in the neighbourhood of Gothenburg, with lunch.

Afternoon: Free in Gothenburg.

Evening: Dinner at Town Hall (Börsen), Gothenburg, by invitation of the firms in Gothenburg to which visits have been made. Members and Ladies will leave Gothenburg by special sleeping-car train for Västerås after dinner.

Wednesday, 9th June (Västerås)

Morning: Members may take part in *one* of the following excursions, with lunch:

Excursion 7—ASEA (Swedish General Electric Co.).
Excursion 8—AB Svenska Metallverken.
Excursion 9—Surahammars Bruks AB.

Ladies may take part in *either*:

Excursion 9—Surahammars Bruks AB (with Members), *or*
Excursion 10—Visit to Västerås and the neighbourhood, with lunch.

Afternoon: Members and Ladies leave by special train for Stockholm.

Evening: Free in Stockholm.

Thursday, 10th June (Stockholm)

Morning: Free in Stockholm.

Mid-day: *Excursion* 11 (Members and Ladies)—Visit to Skansen, with lunch.

Afternoon: Free in Stockholm.

SECTION II—STOCKHOLM

Thursday, 10th June

Evening: Reception at Jernkontoret, afterwards free in Stockholm.

Friday, 11th June

Morning: Members may *either* join the Technical Session of Jernkontoret at Riksdaghuset, *or* have the morning free, *or* take part in *one* of the following excursions with lunch:

Excursion 12—AB Atlas Diesel.
Excursion 13—AB Svenska Gasaccumulator (Aga).
Excursion 14—Telefon AB L. M. Ericsson.
Excursion 15—Svenska Diamantbergborrnings AB (Swedish Diamond Rock Drilling Co.).
Excursion 16—Technical Museum.
Excursion 17—Kungl. Tekniska Högskolan (Royal Institute of Technology) and Metallografiska Institutet (Institute for Metal Research).

Ladies may take part in *either*:

Excursion 16—Technical Museum (with Members), *or*
Excursion 18—Visit to Waldemarsudde and Millesgården, with lunch at Foresta.

Afternoon: Free in Stockholm.

Evening: Special Performance at the Royal Opera House by invitation of Jernkontoret. (Evening dress will be worn, no decorations.)

Saturday, 12th June

Morning: Joint Technical Session of The Iron and Steel Institute and Jernkontoret at Riksdagshuset (*see below*).

Ladies: *Excursion* 19—Visit by bus to Drottningholm Royal Theatre with short ballet performance.

Afternoon: Free in Stockholm.

Evening: Banquet and Dance at the Town Hall by invitation of Jernkontoret. (Evening dress with decorations will be worn.)

Sunday, 13th June

Members and Ladies may take part in *either*:

All day: *Excursion* 20—Visit by motor bus to Uppsala, Skokloster, and Sigtuna, with lunch at Flustret, Uppsala.

or

Morning: Free in Stockholm.

Afternoon: *Excursion* 21—Excursion by boat round the waterways of Stockholm.

(*Note:* Members and Ladies going on Special Train Excursion C: Lapland (*see below*) will not be able to take part in either *Excursions* 20 or 21.)

SECTION III—SPECIAL TRAIN EXCURSIONS

Members and Ladies will be able to take part in *one* of the three alternative Special Train Excursions, as follows:

Special Train Excursion A: Värmland

Sunday, 13th June

Evening: Special train leaves Stockholm.

Monday, 14th June

Morning: Special train arrives at Kopparberg. Members and Ladies will be the guests of Hellefors Bruks AB throughout the day.

Excursion 22 (Members and Ladies)—Bångbro Cold Rolling Mills.

Mid-day: Special train arrives at Hellefors. Lunch at Hellefors.

Afternoon: *Excursion* 23 (Members)—Hellefors Iron and Steel Works. *Excursion* 24 (Ladies)—Visit in the neighbourhood of Hellefors.

Evening: Dinner at Saxåhyttan.

Tuesday, 15th June

Morning: Special train arrives at Filipstad. Members and Ladies will be the guests of the Uddeholms AB during this and on the following day until leaving Karlstad.

All day: Members may take part in *one* of the following:

Excursion 25—Storfors Tube Works (morning) and Persberg Iron Mines and Sponge Iron Plant (afternoon).

Excursion 26—Persberg Iron Mines and Sponge Iron Plant (morning) and Hagfors Iron and Steel Works (afternoon).

Excursion 27—Storfors Tube Works (morning) and Hagfors Iron and Steel Works (afternoon).

Ladies: *Excursion* 28—Visit to Wasa Spisbrödsfabrik and other places of interest in Filipstad and the neighbourhood.

Members and Ladies will have lunch at Spångberget.

Evening: Dinner at Uddeholm, then return by bus to special train at Filipstad.

Wednesday, 16th June

Morning: Special train arrives at Karlstad. Members may take part in *either*:

Excursion 29—Skoghall Works, with lunch, *or*

Excursion 30—Lake Fryken, Rottneros, and Mårbacka, with lunch.

Ladies may take part in *Excursion* 30 with Members.

Afternoon: Special train leaves Karlstad and arrives at Bofors. Members and Ladies will be the guests of AB Bofors till leaving Bofors on the following day.

Evening: Dinner at Bofors.

Thursday, 17th June

Morning: *Excursion* 31 (Members)—Bofors Steel Works.

Excursion 32 (Ladies)—Visits to the neighbourhood of Bofors.

Afternoon: Special train leaves Bofors, arriving at Gothenburg the same evening, for connection with the mail boat service to the U.K.

(*Note:* Members and Ladies not returning to England on 17th June will be able to leave the special train at Laxå and proceed by train direct to Stockholm, arriving the same evening.)

Special Train Excursion B: Dalecarlia

Sunday, 13th June

Evening: Special train leaves Stockholm.

Monday, 14th June

Morning: Special train arrives at Skutskär. *Excursion* 33 (Members and Ladies)—Skutskär Works (Stora Kopparbergs Bergslags AB), with lunch at the Works.

Afternoon: Special train leaves Skutskär, arriving at Sandviken.

Excursion 34 (Members)—Sandviken Iron and Steel Works.

Excursion 35 (Ladies)—Visits in the neighbourhood of Sandviken.

Evening: Dinner at Sandviken by invitation of the Sandvikens Jernverks AB.

Tuesday, 15th June

Morning: Special train leaves Sandviken, arriving at Borlänge. Members and Ladies will be the guests of Stora Kopparbergs Bergslags AB on this day and during the morning of 16th June, until leaving Leksand.

Excursion 36 (Members)—Domnarfvet Iron and Steel Works, with lunch.

Excursion 37 (Ladies)—Visits in the neighbourhood of Domnarfvet, with lunch.

Afternoon: Travel by bus from Domnarfvet to Falun.

Excursion 38 (Members and Ladies)—Visits to Falun Copper Mine and Bergslager Museum.

Evening: Dinner at Bergslagssalongen at the Headquarters of Stora Kopparbergs Bergslags AB.

Wednesday, 16th June

Morning: Special train leaves Falun, arriving at Mora.

Excursion 39 (Members and Ladies)—Sightseeing by bus. Mora (Zorn Museum), Rättvik, Tällberg (for lunch), and Leksand.

Afternoon: Special train leaves Leksand, arriving at Fagersta. Members and Ladies will be the guests of Fagersta Bruks AB until leaving Fagersta on the following day.

Evening: Dinner at Fagersta.

Thursday, 17th June

Morning: *Excursion* 40 (Members)—Fagersta Iron and Steel Works. *Excursion* 41 (Ladies)—Visits in the neighbourhood of Fagersta.

Afternoon: Special train leaves Fagersta for Gothenburg, arriving the same evening for connection with the mail boat service to the U.K.

(*Note:* Members and Ladies not returning to England on 17th June will be able to go by train direct to Stockholm, arriving the same evening.)

Special Train Excursion C: Lapland

Sunday, 13th June

Morning: Special train leaves Stockholm; all day on the train.

Monday, 14th June

Mid-day: Special train arrives at Narvik. Members and Ladies will be the guests of Luossavaara-Kiirunavaara AB on 14th, 15th, and 16th June, until leaving Malmberget.

Afternoon: *Excursion* 42 (Members and Ladies)—Visit to Narvik Harbour and iron ore loading installations, with lunch.

Evening: Special train arrives at Abisko. Dinner at Abisko Tourist Hotel. The midnight sun can be seen in fine weather.

Tuesday, 15th June

Morning: Special train leaves Abisko, arriving at Kiruna.

Excursion 43 (Members and Ladies)—Kiruna Iron Mines, with lunch.

Evening: Special train leaves Kiruna, arriving at Malmberget. Dinner.

Wednesday, 16th June

Morning: Members and Ladies may take part in *either: Excursion* 44—Malmberget Iron Mine, *or Excursion* 45—Harsprånget Power Plant.

Mid-day: Special train leaves Malmberget and arrives at Luleå.

Afternoon: *Excursion* 46 (Members and Ladies)—Norrbotten Iron and Steel Works.

Evening: Dinner by invitation of Norrbottens Järnverk AB.

Thursday, 17th June

Morning: Special train arrives at Rönnskär. Members and Ladies will be the guests of Bolidens Gruv AB.

Excursion 47 (Members and Ladies)—Rönnskär Smelting Works, with lunch.

Afternoon: *Excursion* 48 (Members and Ladies)—Boliden Copper Mines.

Evening: Special train leaves Bastuträsk.

Friday, 18th June

Morning: Special train arrives at Stockholm.

Technical Sessions in Stockholm

A Technical Session of Jernkontoret will be held at the Riksdagshuset, Stockholm, on Friday, 11th June, 1954, commencing at 10.0 A.M. Jernkontoret have extended a cordial invitation to any Members of The Iron and Steel Institute who are interested to attend. The proceedings will be in Swedish, but contributions to the discussion in English will be welcomed. The following papers, which are published in this issue of the *Journal*, will be presented for discussion:

"*Aspects of Swedish Iron Ore Concentration*," by P. G. Kihlstedt.

"*Calculation of Roll Pressure and Energy Consumption in Hot Rolling*," by G. Wallquist

"*Deep-Drawing Properties of Sheet Steel: Fundamental Principles and Test Methods*," by O. E. Svahn.

A Joint Technical Session of The Iron and Steel Institute and Jernkontoret will be held at the Riksdagshuset on the morning of Saturday, 12th June, 1954. The proceedings will be in English. The following papers which are published in this issue of the *Journal*, will be presented for discussion:

10.0–11.45 A.M.—Joint Discussion on:

"*Aspects on Pelletizing of Iron Ore Concentrates*," by Magnus Tigerschiöld

"*The Development of a Pelletizing Process for Fine Iron Ores*," by J. M. Ridgion, E. Cohen, and C. Lang

"*The Pelletizing of Northampton Sand Ironstones by Vacuum Extrusion: The Experimental and Pilot Plants at the Corby Works of Stewarts and Lloyds Ltd.*," by A. Stirling

11.45–11.50 A.M.—The following paper will be presented but not discussed:

"*The Universal Decimal Classification Applied to Metallurgical Literature*," by E. Öhman and J. P. Saville

11.50 A.M.–1.0 P.M.—Joint Discussion on:

"*Tensile and Impact Properties of Fe–Si, Fe–Ni, Fe–Cr, and Fe–Mo Alloys of High Purity*," by W. P. Rees, B. E. Hopkins, and H. R. Tipler

"*Effect of Heat-Treatment on the Brittleness of High-Purity Iron–Nitrogen Alloys*," by B. E. Hopkins and H. R. Tipler

"*Pearlite-Free Basic Bessemer Steel: Its Fabrication and Properties*," by A. Josefsson

The following papers prepared in connection with the Meeting are also published in this issue of the *Journal*. They will not, however, be formally presented for discussion:

"*Iron and Steel Making Processes used in Sweden*," by S. Fornander

"*Reduction of Iron Ore Without Melting in a Rotary Furnace*," by B. Kalling and F. Johansson

"*Formation of Martensite in Austenitic Stainless Steels: Effects of Deformation, Temperature, and Composition*," by T. Angel

"*Practical Aspects of Hardness Testing of Steel Strip*," by A. G. Molinder

"*A Gravimetric Method of Gas Analysis*," by J. Morsing

"*A Theory of Hydrogen Embrittlement*," by F. de Kazinczy

Synopses of other officially prepared papers published in earlier issues of the *Journal* are given for reference on pp. 189–190.

JERNKONTORET

Jernkontoret is the central organization of the Swedish Iron and Steel Industry and represents the industry in all questions of common interest except labour relations. It speaks for the iron and steel industry in its relations with the Government and is frequently asked to submit reports and information on questions relating to the industry. The expert knowledge of Jernkontoret is utilized by the Government in negotiations for trade agreements with foreign countries and in deliberations within O.E.E.C. and E.C.E. committees, etc.

At the time of the foundation of Jernkontoret in 1747, Sweden was the largest iron producer in the world. The industry consisted of a large number of small blast-furnaces and forges, economically weak and often in the hands of large exporting firms, which furnished capital for the ironworks but took advantage of their strong position to depress iron prices. The first aim of Jernkontoret was the financial strengthening of the iron market.

From the outset, Jernkontoret was also actively engaged in the scientific and technical development of the industry. In 1751 the famous metallurgist Sven Rinman was appointed 'Smelting Master.' Since that time, Jernkontoret has always had a staff of technical specialists and has contributed actively to the introduction and improvement of new technical devices, methods, and processes. An internationally famous member of the technical staff in more recent times was J. A. Brinell.

The need to lower production costs, to investigate new processes and methods, and to develop and study the great variety of steels required by modern techniques were incitements to the establishment at Jernkontoret in 1926 of a research organization on modern lines. This

is financed by special contributions from the participating works. The annual research budget at present totals about £75,000. Of this sum, a considerable proportion goes to Metallografiska Institutet (the Institute for Metal Research) as a grant for fundamental research on metals and alloys. Closely associated with Jernkontoret's research work on metallurgical and allied questions is the research conducted by Svenska Gruvföreningen (the Swedish Mining Association) on mining and ore dressing problems, which is supported by all Swedish mines including non-members of Jernkontoret. The annual budget for this research work is about £14,000.

The actual research work is entrusted to small research sub-committees, mainly working at industrial laboratories or in works' departments. Many investigations are also carried out at Metallografiska Institutet and in the various departments of the Royal Institute of Technology in Stockholm.

The Research Department publishes part of the results as internal confidential reports, but in the main they are published in *Jernkontorets Annaler*, a monthly journal which has been published regularly since 1817. Jernkontoret also has a well-equipped library specializing in metallurgical and mining literature, combined with an information department which edits an abstracting journal, *Jernkontorets Litteraturöversikt*.

Since the early nineteenth century, Jernkontoret has shown an interest in technical training. Between about 1820 and 1860, the School of Mines at Falun was mainly financed by Jernkontoret. Its successor, the Lower School of Mines at Filipstad, is run largely at the expense of Jernkontoret; grants are also given to cover the costs of certain practical training courses at the School of Mines at the Royal Institute of Technology.

The centre of the activities of the organization is the Jernkontoret building at Kungsträdgårdsgatan 6, Stockholm. The building contains an assembly hall and a number of committee rooms and is the natural focal point for metallurgists and mining experts.

Council and Officers of Jernkontoret

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EVERT WIJKANDER	AB Bofors

Members of Council

HÅKAN ABENIUS	Stora Kopparbergs Bergslags AB
EINAR AMÉEN	Surahammars Bruks AB
BENGT CARLESON	Degerfors Järnverks AB
NILS DANIELSEN	Uddeholms AB
THORSTEN EKSTRAND	AB Garphytte Bruk
ERIK W. FORSBERG	Sandvikens Jernverks AB
HILDING JOHANSSON	Norrbottens Järnverk AB
ERIK KOCKUM	Kockums Jernverks AB
JOEL LARSSON	AB Svenska Kullagerfabriken
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HÅKAN DAHLERUS—Director of Economic Affairs
SVEN FORNANDER—Director of Research
EINAR ÖHMAN—Head of Publication and Information Department
KARL FREDRIK LINDSTRAND—Senior Assistant to the Director of Research

Responsible for the Meeting Organization

GÖSTA FRISELL—formerly Managing Director of Jernkontoret

BIOGRAPHIES

Jernkontoret

Evert Wijkander was born at Ramen, Sweden, in 1891. He received his technical education at the Royal Institute of Technology, Stockholm, graduating as a metallurgical engineer in 1912. In 1914 he went to the U.S.A. for two years, where he worked in the Homestead Steelworks, Pittsburgh, and then spent a year in Germany.

Mr. Wijkander's first industrial appointment was as Assistant Melting-Shop Manager with AB Bofors in 1918. Two years later he was appointed Melting-Shop Manager at the Hagfors Steelworks, and in 1924 he became Works Manager of Fagersta Bruks AB, a position which he held for ten years. In 1935 he rejoined the Bofors concern, and a year later was appointed Managing Director, a position which he still holds. He is also President of the Board of Directors of a number of subsidiary companies, and a member of the Boards of several other companies.

Since 1945 Mr. Wijkander has been President of the Swedish Iron and Steel Works Employers' Association; he has also been President of the Swedish Institute for Metal Research since 1949. He has taken an active part in the work of other industrial organizations and is a Member of Council of the Federation of Swedish Industries and of the Swedish Employers' Confederation. Until recently he was also a Member of Council of the Federation of Swedish Mechanical Engineering Industries. He is a member of the Royal Swedish Academy of Military Science, the Swedish Academy of Engineering Sciences, and the Swedish Academy of Naval Science.

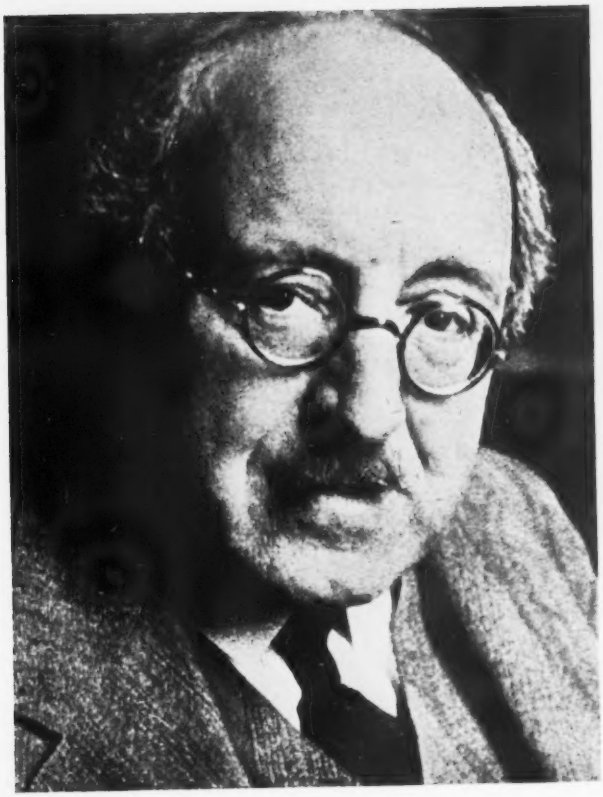
Mr. Wijkander became a Member of Council of Jernkontoret in 1938, and was elected President in 1952. He became a Member of the Research Council of Jernkontoret in 1933, and served as President of the Council from 1948 until 1952.

Gösta Frisell was born at Falun, Sweden, in 1885. On leaving school he joined the regular army and received his first commission in 1905. He graduated from the Staff College and in 1917 became a captain on the Swedish General Staff. He was appointed major in the Swedish Army in 1935.

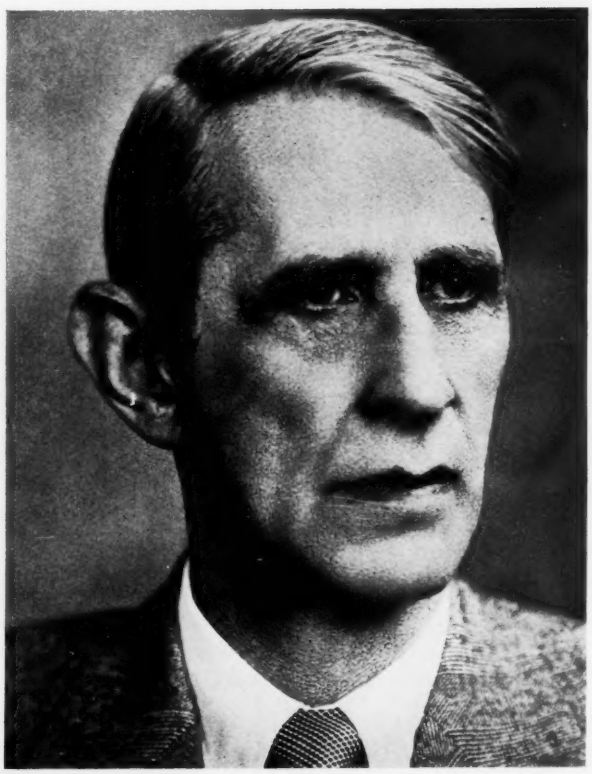
Mr. Frisell joined the reserve in 1919 to become a Director of Stora Kopparbergs Bergslags AB. He served as a "deputerad" (governing trustee) of Jernkontoret from 1926 to 1945, and on leaving the Company in 1946 he became Managing Director of Jernkontoret, a position which he held until his retirement in 1953. During this time he was a Member of Council of Jernkontoret, and in 1953 he was commissioned by the Council to take charge of preparations for the Special Meeting of The Iron and Steel Institute in Sweden.

Mr. Frisell was President of the Swedish State Transport Commission from 1944 to 1950, and since 1947 he has been a member of the Swedish Railway Council. He is also a member of the Recruitment Commission of the Royal Swedish Ministry of Foreign Affairs. He is a member of the Swedish permanent delegation at the High Authority of the European Coal and Steel Community in Luxembourg, and is the Swedish delegate on steel committees within E.C.E. and O.E.E.C. Mr. Frisell is also a member of the Royal Swedish Academy of Military Science.

Ragnar Sundén was born at Umeå, Sweden, in 1907. He graduated from the Stockholm School of Economics



CARL BENEDICKS
Bessemer Medallist 1927



MAGNUS TIGERSCHIÖLD
Bessemer Medallist 1947

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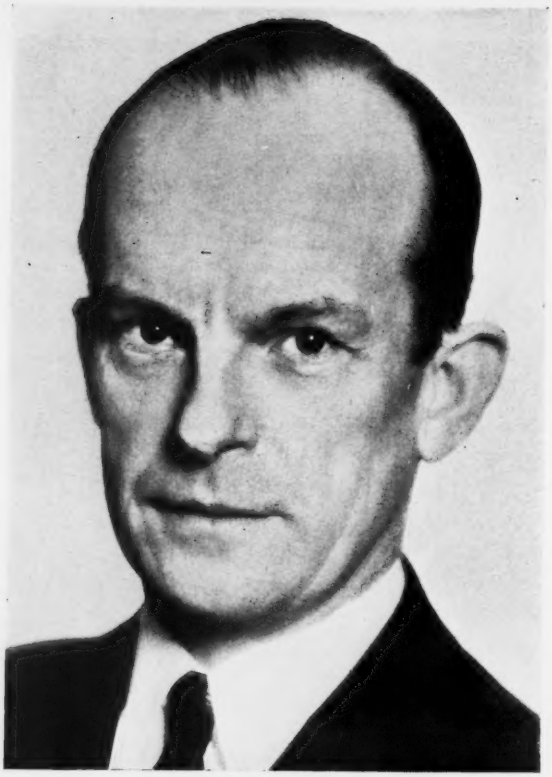
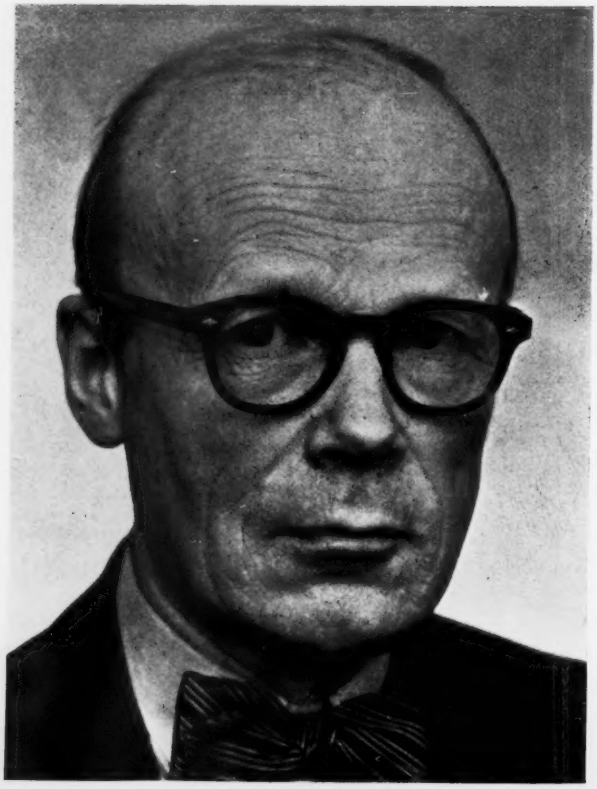


HÅKAN DAHLERUS
Director of Economic Affairs of Jernkontoret

SVEN FORNANDER
Director of Research of Jernkontoret



EINAR ÖHMAN
Head of Publication and Information Department, Jernkontoret

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in 1929, and after economic studies abroad took up his studies in the Law Faculty of Stockholm University, graduating in 1935.

In the following year, Mr. Sundén joined the staff of the Swedish Ministry of Finance, serving as Assistant to the Under-Secretary of State, Mr. Dag Hammarskjöld, now General Secretary of the United Nations. In 1939 he was given charge of the organization of the Swedish Institute for Industrial, Economic, and Social Research, and subsequently became the first head of this Institute. In 1941, he was appointed Under-Secretary of State for Culture and Education. During this time he also initiated the Swedish National Science Research Council.

In 1945 Mr. Sundén became Director in charge of financial and international affairs at the Federation of Swedish Industries, and later held the posts of Advisor on financial administration to the Venezualan Government (1948), and Chairman of the United Nations Economic Mission in Ecuador (1949–50). He served as Consultant to the United Nations on the organization of the technical aid programme for Bolivia in 1953. In the same year he was appointed Managing Director of Jernkontoret, a position which he still holds.

Mr. Sundén has been an alternate member of the Board of Directors of the Central Bank of Sweden since 1947, and for six years he served on the Board of Directors of the Swedish National Broadcasting Company. He has been a member of the Board of the Swedish Institute for Cultural Relations with Foreign Countries since 1945, and since 1947 he has served as Vice-Chairman.

Håkan Dahlerus was born in 1898, and received his technical education at the Royal Institute of Technology, Stockholm, graduating in 1924. In the following year he joined the staff of Jernkontoret, serving first as Assistant to the chief Technical Advisor, and from 1928 as Senior Assistant to the Director of Research. In 1930 he became Assignee and Secretary to the Board of Jernkontoret, and was appointed Director in 1944.

Mr. Dahlerus has been Head of the Iron and Steel Department of the Swedish State Commission for Trade and Industry since 1950, and for a number of years he has served as a Technical Expert to the Royal Swedish Ministry of Foreign Affairs.

Sven Fornander was born at Hagfors, Sweden, in 1913. He received his technical education at the Royal Institute of Technology, Stockholm, graduating as a metallurgical engineer in 1939.

His first appointment was with the Swedish Institute for Metal Research, where he worked as research assistant under the late Dr. Gösta Phragmén. From 1941 to 1951 he was with Surahammars Bruks AB, first as Assistant Melting-Shop Manager, and later as Research Manager. He took up his present appointment as Director of Research of Jernkontoret in 1952.

Mr. Fornander is the author of several papers dealing with temperature measurement in the steel bath, theoretical and practical investigations on the basic open-hearth process, inductive stirring in arc furnaces, and the desulphurization of pig iron.

Einar Öhman was born in 1896, and was educated at the Royal Institute of Technology, Stockholm, graduating in 1921. In the following year he took up an appointment on the teaching staff of the Lutheran College in Yiyang, China. Returning to Sweden, he entered Stockholm University, where he carried out a programme of X-ray crystallographic research mainly on the iron–manganese system and on martensite, under Professor Arne Westgren, and where he held the position of Demonstrator in Chemistry from 1928 to 1930. He became Assistant Editor of *Jernkontorets Annaler* in 1931, and four years later was appointed Editor. He also acted as Reader in General Metallurgy at the Royal Institute of Technology from 1931 to 1938.

Mr. Öhman then joined the Bofors concern, first as a research metallurgist at the Bofors steelworks, and later, from 1942, as Chief Metallurgist at the light metal works of AB W. Dan Bergman at Weda. In 1948 he returned to Jernkontoret as Head of the Information Department and the Library, and two years later he was in addition appointed Editor of *Jernkontorets Annaler*. He also held the post of Reader in X-ray Crystallography at the Royal Institute of Technology from 1948 to 1952.

Mr. Öhman is President of the Swedish Society for Technical Documentation, and is actively engaged in work on the revision of the Universal Decimal Classification within the International Federation of Documentation.

Swedish Bessemer Medallists

Carl Axel Fredrick Benedicks was born in Stockholm in 1875. He was educated at the Beskowska School and at the University of Uppsala, where he was awarded the degrees of Fil. Lic. (1902), and Fil. Dr. (1904). He holds the honorary degrees of Dr. Ing. e.h. from Charlottenburg and Dr. Techn. h.c. from Cracow.

Professor Benedicks began his career as Lecturer in Physical Chemistry at the University of Uppsala in 1904. In 1911 he was appointed Professor of Physics at the University of Stockholm, and from 1920 to 1935 he served as the first Director of Metallografiska Institutet—an institute for metallographic research which was founded on his initiative. Since 1935 he has been engaged in research at his private laboratory in Stockholm.

The scientific activities in which Professor Benedicks has been, and is, engaged cover a wide field. Although his reputation is based solidly on work on the structure and chemistry of metals, his interests and the experiments that he has carried out are by no means restricted to these subjects. He is a skilled experimenter, and has devoted much effort to the development of new and improved laboratory techniques. His papers on metals are concerned with problems in electrical conductivity, thermo-electric phenomena, mechanical properties, phase transformations, equilibrium diagrams, especially of the iron–carbon system, and the nature of bainite.

Professor Benedicks enjoys an international reputation. He has been awarded the Bergstedt Prize (1902), the Edlund Prize (1904), the Arnberg Prize (1919), the Prix Henry Wild (1922), and the Prix Becquerel (1936). He is a member of many technical societies in Great Britain and other countries, and is a Past-President of the Metals Section of the International Association for Testing Materials.

Professor Benedicks became a Member of The Iron and Steel Institute in 1905, and was appointed an Honorary Vice-President in 1922. He was awarded the Carnegie Gold Medal in 1908 and the Bessemer Gold Medal in 1927.

Magnus Tigerschiöld was born in Stockholm in 1893. He studied metallurgy at the Royal Institute of Technology, Stockholm, graduating as a metallurgical engineer in 1917.

His first industrial experience was obtained at Trollhättan, but he left that concern after a short time to become assistant to the General Superintendent at the Söderfors steelworks. In 1925 he was appointed Superintendent of the open-hearth plant at Fagersta Bruks AB, and two years later became Superintendent in charge of research for the whole Fagersta combine. In 1936 he was appointed Director of Research of Jernkontoret, in Stockholm, where he was responsible for developing and maintaining relations with corresponding

foreign institutions. In 1951 Dr. Tigerschiöld left Jernkontoret to take up his present appointment as Director of Metallurgical Research and Development to the Trafikaktiebolaget Grängesberg-Oxelösund and Luossavaara-Kiirunavaara AB.

Dr. Tigerschiöld's work has been devoted mainly to the development of methods of manufacturing pig iron and steel, and he is the author of numerous papers on these and related subjects. He has been a member of the Royal Swedish Academy of Engineering Sciences since 1935, and a member of the Swedish State Council of Technical Research since 1943. In 1945 he was awarded the degree of Doctor honoris causa of the University of Uppsala; he is also a recipient of the Rinman Medal of Jernkontoret and of the Carl Lueg Medal of Verein deutsche Eisenhüttenleute.

He has been a Member of The Iron and Steel Institute since 1926, and in 1947 he was awarded the Bessemer Gold Medal.

Contributors to the Journal

Tryggve Angel–Research Associate with the Sandviken Steel Works Co., Ltd., Sandviken, Sweden.

Mr. Angel was born in 1921. He received his technical education at the Royal Institute of Technology at Stockholm, graduating in 1946. After four years as a research engineer in the field of stainless steels, he obtained a Fellowship from the Sweden-America Foundation and spent a year in post-graduate studies at the Massachusetts Institute of Technology, U.S.A. He then joined the Sandviken Steel Works Co., Ltd., where he is now Division Head at the Central Laboratory.

E. Cohen, M.Sc., F.G.S.—Lecturer in Applied Mineralogy at the Royal School of Mines.

Mr. Cohen was born in Vienna in 1920. He was educated at Birkbeck College and Chelsea Polytechnic, graduating with Honours in Geology in 1948. In the same year he joined the Ironmaking Division of the British Iron and Steel Research Association as a geologist, working on problems of ore beneficiation and sintering. As Senior Scientific Officer he was partly responsible for the development of B.I.S.R.A.'s pelletizing project. At the same time he held the post of part-time teacher of Geology at Chelsea Polytechnic. He took up his present appointment in 1953, and has since been teaching and carrying out research in mineral dressing. He continues to be interested in the treatment of iron ores, and is a member of the Burden Committee of B.I.S.R.A.

Folke Johansson—Assistant to the Director of Research, Stora Kopparbergs Bergslags AB, Domnarvet, Sweden.

Mr. Johansson was born in 1914. He studied metallurgy at the Royal Institute of Technology, Stockholm, graduating in 1939. In the same year he was engaged by Sandviken Iron and Steel Works, where he was appointed Superintendent of the electric steel plant in 1942. He took up his present appointment in 1948. His work is concerned mainly with research on the metallurgy of iron and steel.

Åke Josefsson—Assistant to the Director of Research, Stora Kopparbergs Bergslags AB, Domnarvet, Sweden.

Mr. Josefsson was born in 1917. He studied metallurgy at the Royal Institute of Technology, Stockholm, graduating in 1939. From 1940 to 1946 he was with Svenska Metallverken, Finspong, where he was engaged mainly in research on non-ferrous metals. He took up his present appointment in 1946, and has since been concerned with research on the properties of mild steels with particular reference to ductility as affected by non-metallic impurities.

Bo M. S. Kalling—Director of Research and Development at Stora Kopparbergs Bergslags AB, Domnarvet, Sweden.

Professor Kalling was born at Djursholm, Sweden, in 1892. He received his technical education at the Royal Institute of Technology, Stockholm, graduating in 1915. From 1916 to 1931 he held various appointments as a metallurgist with Domnarfvets Jernverk, Domnarvet, the Ferrolegeringar Co., Trollhättan, and the Avesta Iron and Steel Works. He was then appointed Professor of Metallurgy at the Royal Institute of Technology, a position which he held until 1940, when he was appointed Technical Director to the Boliden Mining and Metallurgical Company. He took up his present appointment in 1945.

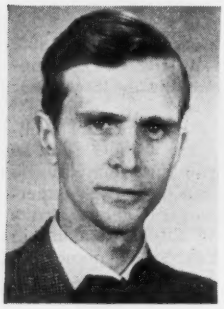
F. de Kazinczy—Physical Metallurgist at the Swedish Institute for Metal Research, Stockholm.

Mr. de Kazinczy was born at Budapest in 1929. He was educated in Denmark from 1945 to 1947, and then went to the Royal Institute of Technology in Stockholm, where he studied metallurgical engineering and graduated in 1953. He took up his present appointment in the same year.

P. G. Kihlstedt—Professor in Mineral Dressing at the Royal Institute of Technology, Stockholm.

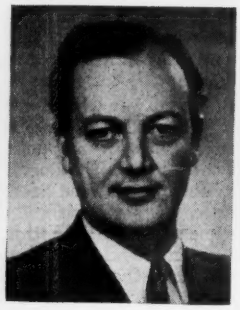
Professor Kihlstedt was born at Örebro, Sweden, in 1909. He received his technical education at the Royal Institute of Technology, Stockholm, where he graduated as a mining engineer in 1933. After a short time in prospecting, he joined the Boliden Mining Company as superintendent at the Rönnskär flotation plant. In 1945 he moved to the Company's Central Organization at Boliden, where he was appointed General Superintendent in 1950. He took up his present appointment in 1952.

A. G. Molinder, M.S.—Chief Metallurgist, and head of the laboratory at Uddeholms AB, Munkfors Bruk, Sweden.

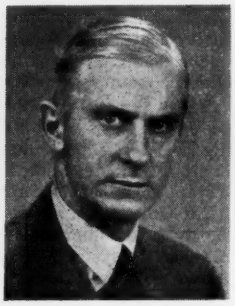


T. Angel

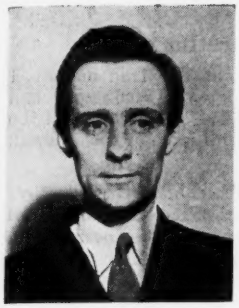
E. Cohen



A. Josefsson



B. Kalling



A. G. Molinder

F. Johansson

F. de Kazinczy

P. J. G. Morsing

Mr. Molinder was born in Stockholm in 1919, and received his technical education at the Royal Institute of Technology. He spent four years carrying out research in the Physical Laboratory at Munkfors, until he was awarded a one-year scholarship to Massachusetts Institute of Technology by the Uddeholm Co. While at M.I.T. he was awarded the degree of M.S. in Metallurgy. He took up his present appointment in 1949.

P. Johan G. Morsing—Late Chief Engineer in the Department of Inorganic Chemistry of the Government Testing Institute, Stockholm, now retired.

Mr. Morsing was born at Wiby, Örebro, Sweden, in 1884. He received his technical education at the Royal Institute of Technology in Stockholm, and in 1908 he was awarded a scholarship from Jernkontoret for study in Germany. A year later he joined Gullspångs Elektrokemiska AB as a chemist, a position which he held until 1912 when he took up an appointment at the Testing Institute of the Royal Institute of Technology, remaining with the Institute when, in 1920, it was reorganized as the Government Testing Institute.

J. M. Ridgion, B.Sc., A.R.C.S.—Principal Scientific Officer in the Ironmaking Division of the British Iron and Steel Research Association.

Mr. Ridgion was born in 1912 and received his technical education at the Royal College of Science, London, graduating with Honours in Chemistry in 1933. After carrying out research in physical chemistry at Armstrong College, Newcastle-on-Tyne, he joined the Technical Department of the British Iron and Steel Federation (I.S.I.R.C.) in 1936, where he acted as Secretary of Technical Committees. He has been with the Ironmaking Division of B.I.S.R.A. since 1945, except for a short period in 1947–48 when he was temporarily seconded to the British Commonwealth Scientific Office in Washington, D.C.

A. Stirling, B.Sc., A.R.T.C., F.I.M.—Manager of the Blast-Furnace Burden Development Section, Department of Research and Technical Development, Stewarts and Lloyds Ltd.

Mr. Stirling was born in 1912, and was educated at the Glasgow Academy, the University of Glasgow, and the Royal Technical College. In 1935 he joined the research staff of Stewarts and Lloyds Ltd., and from 1937 to 1942 he worked in the Fuels and Furnaces Section of the Company at Bilston. He was then seconded to the Ministry of Supply as Technical Adviser to the Directorate of Thermal Engineering. He rejoined Stewarts and Lloyds Ltd. after the war, and was appointed Technical Assistant to the Managing Director of Iron and Steel Production. He took up his present appointment at Corby in 1951.

Mr. Stirling is a member of the Blast-Furnace Practice and Process Committees and the Burden Committee of B.I.S.R.A. He was elected a Member of The Iron and Steel Institute in 1934.

Olov E. Svahn—Production Manager of the Sandviken Steel Works, Sandviken, Sweden.

Dr. Svahn was born in 1914. He received his technical education at the Royal Institute of Technology, Stockholm, where he graduated in mechanical engineering in 1939, and where he was awarded the degrees of Licentiate of Technology (1948) and Doctor of Technology (1949). Remaining at the Institute he worked first as a research engineer in the department of mechanical technology of the Institution of Machining and Metal Working. He became Assistant in the Institution, and later was made acting Professor. During this time he served as consulting research engineer to a number of Swedish and foreign industrial concerns. In 1944 he joined the Sandviken Steel Works, where he was responsible for the organization of the machinability research department. He became head of the department in 1948, and took up his present appointment four years later.

Dr. Svahn has studied machinability problems, with particular reference to sintered carbides, and has carried

P. G. Kihlstedt

J. M. Ridgion

A. Stirling

O. E. Svahn

out research on milling problems, cutting fluids, and deep-drawing. He is the author of many papers on these subjects.

J. P. Saville, B.Sc.(Hons.)—Information Officer to The Iron and Steel Institute.

Mr. Saville was born at Sutton Coldfield in 1911. He was educated at Handsworth Grammar School and at the University of Birmingham, where he was awarded the degree of B.Sc.(Hons.) in Metallurgy in 1932. After a short period of research work he was appointed Assistant Assayer at Taquah and Abosso Gold Mines, Gold Coast Colony. Returning to the U.K. in 1934, he joined Murex Ltd. as a Works Chemist. In 1940 he took up a post in the Armament Research Department of the Ministry of Supply; at the end of the war he was seconded to the British Intelligence Objectives Sub-Committee, and was stationed at H.Q., B.A.O.R. He joined the staff of The Iron and Steel Institute as Assistant Information Officer in 1947, and was appointed Information Officer on 1st January, 1954.

Gunnar Wallquist—Professor of Working and Treating of Steel and Dean of the Department of Mining and Metallurgy at the Royal Institute of Technology, Stockholm.

G. Wallquist

Professor Wallquist was born at Steneby, Sweden, in 1894, and received his technical education at the Royal Institute of Technology, graduating in 1917. He worked for different Swedish steel companies until 1929, when he took up his present appointment.

Professor Wallquist's main interest has been in the field of hot rolling of steel. Most of his published papers deal with rolling problems, but some are concerned with plasticity, treatment, and testing of steel. For a number of years he has been a consultant to the Swedish steel industry and to steelworks abroad on hot-rolling questions. He is a member of various Jernkontoret Permanent Research Committees, and of the Swedish Academy of Engineering Sciences.

B. E. Hopkins, M.Sc.—Principal Scientific Officer in the Metallurgy Division of the National Physical Laboratory, Teddington.

Mr. Hopkins took an Honours degree in Metallurgy at University College, Swansea, in 1939. He then joined the Research Laboratory of the Mond Nickel Co., Ltd., Birmingham, where he worked on alloy steels and cast irons. In 1946 he was awarded the M.Sc. degree of the University of Wales. A year later he joined the National Physical Laboratory, and has since been working on the effects of alloying elements on the transition from tough to brittle fracture in high-purity iron. During 1948 Mr. Hopkins was seconded as a liaison officer to the British Commonwealth Scientific Office, Washington, D.C.

H. R. Tipler, B.Met.—Research worker in the Metallurgy Division of the National Physical Laboratory, Teddington.

Mr. Tipler was educated at High Storrs Grammar School, Sheffield, and Sheffield University, where he graduated in 1947. He then took up his present appointment at the National Physical Laboratory, and has since been engaged on an investigation into the effects of alloying elements on the incidence of brittle fracture in high-purity iron.

INSTITUTE NEWS

Annual General Meeting, 1954

The **Annual General Meeting** of The Iron and Steel Institute will be held in London on **Wednesday and Thursday, 26th and 27th May, 1954.** Full details of the programme were given in the April issue of the *Journal*.

Autumn General Meeting

The **Autumn General Meeting** of The Iron and Steel Institute will be held in London on **Wednesday and Thursday, 24th and 25th November, 1954.**

Symposium on Powder Metallurgy, 1954

A **Symposium on Powder Metallurgy** is being organized by The Iron and Steel Institute, in association with the Institute of Metals, to be held at Church House, Westminster, London, S.W.1, probably on **1st and 2nd December, 1954.** An organizing Committee has been set up as follows:

Chairman

Dr. Ivor JENKINS	General Electric Co., Ltd.

Members

Mr. L. J. BRICE	Ministry of Supply
Dr. W. D. JONES	Consultant
Mr. D. A. OLIVER, C.B.E.	B.S.A. Group Research Centre
Dr. L. B. PFEIL, O.B.E., F.R.S.	Mond Nickel Co., Ltd.
Dr. T. RAINE	Metropolitan-Vickers Electrical Co., Ltd.
Mr. E. ROBSON	Manganese Bronze and Brass Co., Ltd.

Secretary

Mr. K. HEADLAM-MORLEY The Iron and Steel Institute

The Symposium will cover both ferrous and non-ferrous aspects of powder metallurgy, and a small exhibition of powder metallurgy components will also be held. Many papers are being specially written for the Symposium, and will be presented and discussed in the following three main groups: Metal Powders and their Assessment; Research and Production Practice; Production and Properties of Engineering Materials. A circular giving further particulars of the meeting will be issued later.

Ablett Prize

The Ablett Prize for 1953 has been awarded to Mr. **F. Starkey** (Buckley and Taylor Ltd.) for his paper on "*Guides and Strippers for Modern Rod and Bar Mills.*"

NEWS OF MEMBERS

▶ Mr. K. V. AIYER has joined the Associated Cement Companies, Ltd., Madukkarai, India.

▶ Mr. W. BAILEY has left Davy and United Engineering Co., Ltd., to take up the appointment of Chief Engineer with the Briton Ferry Steel Co., Ltd.

▶ Sir JOHN DUNCANSON, an Honorary Vice-President of the Institute, has joined the Board of the Lancashire Steel Corporation Ltd.

▶ Mr. H. T. EVANS has left the Deloro Smelting and Refinery Co., Ltd., to become Supervisor of Research to the Nickel Processing Corporation of New York.

▶ Mr. J. J. EVANS has left Richard Thomas and Baldwins Ltd. to become Assistant Blast-Furnace Manager with John Summers and Sons Ltd.

▶ Mr. F. GARTSIDE has left the United Steel Companies Ltd. to take up an appointment as Technical Manager with the Tempered Spring Co., Ltd., Sheffield.

▶ Mr. J. C. HOWARD, a Director of the Electric Furnace Co., Ltd., has been made a Director of EFCO Engineering Co., Ltd.

▶ Dr. J. P. HUGO has left the South African Iron and Steel Industrial Corporation to take up an appointment in the Metallurgical Section of the Council for Scientific and Industrial Research in Pretoria.

▶ Mr. H. D. JONES has left Dorman, Long and Co., Ltd., to join the Round Oak Steel Works Ltd., Brierley Hill.

▶ Mr. L. KIDMAN has been made an Associate Member of the Institute of Fuel.

▶ Mr. E. C. KLEPP is now with Dowson and Dobson Ltd., Johannesburg, S. Africa.

▶ Mr. A. T. LAWTON is leaving India in June to take up permanent residence in the U.K.

▶ Mr. J. G. MCMASTER has left Thos. Cook and Son Ltd., Vancouver, to join the Norton Company, Niagara Falls, Ontario, Canada.

▶ Mr. S. MARTON has left Sheepbridge Engineering Ltd. to join Sintered Products Ltd. as a metallurgist.

▶ Mr. D. MAXWELL has been appointed Lecturer in Metallurgy at the University of Sheffield.

▶ Mr. D. J. K. PARKES has transferred to the Operational Research Section of the British Iron and Steel Research Association.

▶ Mr. N. B. RUTHERFORD has left the British Non-Ferrous Metals Research Association to join Cam Gears Ltd., Luton, as a metallurgist.

▶ Mr. G. W. STEWART has left Petters Ltd. to join Rotax Ltd., Hemel Hempstead.

▶ Mr. J. VEVERS has left Simon Carves (Africa) Pty. Ltd., to become Manager of John Scott and Co., Johannesburg, S. Africa.

▶ Mr. T. W. WHITING has left the U.K. for Canada, where he has taken up the post of Co-ordinator of Basic Material Practices at the Directorate of Engineering Standards and Naval Specifications, Ottawa.

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

Irons and Steels for Research Purposes

The British Iron and Steel Research Association has established a store of analysed irons and steels for research purposes, and can now supply samples to research workers. The stock comprises samples from over 50 casts of steel, and includes a number of the steels referred to in publications of The Iron and Steel Institute; *e.g.*, all those mentioned in Special Report No. 24 (Second Report of the Alloy Steels Research Committee) are included in the store. In addition, a limited amount of special high-purity iron is available. A test certificate is issued with each sample giving all available data regarding its cast history, mechanical and heat-treatment properties, composition, etc. Users are asked to communicate any additional data obtained in the course of their research. Thus a direct comparison of research results with those of other workers who have used the same materials will be possible.

Full details of this service are given in a catalogue, copies of which will be supplied on request. The service is normally free and not confined to members of the Association. Samples can be sent abroad subject to the prevailing customs regulations, but a charge is usually made in such cases to cover transport and insurance. All enquiries should be marked ' Pedigree Steels ' and sent to the Metallurgy (General) Division, British Iron and Steel Research Association, Hoyle Street, Sheffield, 3.

Physical Society Exhibition

A furnace-scanning periscope for investigating temperature distribution over the roof of an open-hearth furnace was one of B.I.S.R.A.'s exhibits at the Physical Society's Exhibition of scientific instruments and apparatus, held on 8th–13th April, 1954. This periscope allows any point in the furnace to be viewed. Another exhibit was a two-image pyrometer, for use mainly with open-hearth furnaces; this was demonstrated in conjunction with a spherical calibration furnace. The use of electrical eddy currents for the study of metal surfaces was also demonstrated.

AFFILIATED LOCAL SOCIETIES

Ebbw Vale Metallurgical Society

A joint meeting of the Ebbw Vale Metallurgical Society and The Iron and Steel Institute was held in the Lecture Room of Richard Thomas and Baldwins Ltd., Ebbw Vale, on 25th March, 1954. Mr. A. W. Ellis, President of the Society, was in the Chair, and Professor G. H. Rawcliffe (University of Bristol) presented a paper on " *The Life and Work of Oliver Heaviside.*"

NEWS OF SCIENCE AND INDUSTRY

Conference on Unit Processes of Oxidation

A Joint Conference on Unit Processes of Oxidation has been arranged between the Institution of Chemical Engineers, the Chemical Engineering Group of the Society of Chemical Industry, the Chemical Engineering Group of the Koninklijk Instituut van Ingenieurs (Royal Institution of Engineers), and the Section for Chemical Technology of the Koninklijke Nederlandse Chemische Vereniging (Royal Netherlands Chemical Society). The Conference will be held at The Hague on 6th and 7th May, 1954, and will deal particularly with the chemical engineering aspects of unit processes of oxidation. It is hoped that the programme will include papers on the oxidation of aliphatic and aromatic hydrocarbons, and the manufacture of tonnage oxygen.

International Union of Crystallography

The Third General Assembly and International Congress of the International Union of Crystallography will be held in Paris from 21st to 28th July, 1954. There will be Congress communications on the afternoons of 21st, 22nd, 23rd, 26th, and 27th, and on the morning of 24th July, and the programme will include lectures on crystal growth, low-temperature structure determinations, clathrate compounds, and neutron diffraction. There will be two symposia, " *The Mechanism of Phase Transitions in Crystals*," and " *The Location and Function of Hydrogen*," and the Organizing Committee has, in answer to requests, decided to include sections on electron diffraction and clay minerals. Sessions on " *The Teaching of Crystallography* " and " *Crystallographic Data* " will also be held in the course of the Congress. A programme of excursions has been arranged for Saturday and Sunday, 24th and 25th July.

Copies of the Second Circular, giving full details of the Congress, may be obtained from Dr. W. H. Taylor, Crystallographic Laboratory, Cavendish Laboratory, Cambridge.

International Conference on Hot Dip Galvanizing

The Third International Conference on Hot Dip Galvanizing, organized by the Zinc Development Association, will be held from 4th to 9th July, 1954. It is the successor to the First and Second International Conferences held at Copenhagen in 1950 and at Düsseldorf in 1952, respectively. The reception and technical sessions will be held in Oxford between 4th and 7th July; the Conference will then move to London for the

banquet and subsequent works visits. The technical sessions will be devoted to recent developments in galvanizing; the finishing, testing, and after-treatment of galvanized articles; dross formation and fluxes; and productivity and incentives. There will also be sessions specially devoted to topics of interest to general, wire, sheet, and tube galvanizers, and a 'Problems Clinic' will be held on the evening of 6th July. The programme will also include visits to galvanizing works in the London and Birmingham areas, to take place on 8th–9th July. Further particulars of the Conference may be obtained from the Zinc Development Association, Lincoln House, Turl Street, Oxford.

Ironworks Extensions at Appleby-Frodingham

The first furnace of the new ironworks project at the Appleby-Frodingham Steel Co. was blown-in on 1st March, 1954, by Sir Walter Benton Jones, Chairman of the United Steel Companies, Ltd. The largest furnace in the country, it has a hearth diameter of 27 ft. and a working volume of 43,000 cu. ft.; it is equipped with 18 hearth tuyeres and 9 bosh tuyeres. Using a 100% sinter burden, it is hoped to produce outputs of well over 6000 tons per week.

A second new furnace is now under construction and, when completed, will be the largest in Europe. It is expected that when both furnaces are in operation, South Ironworks, which will then be a 4-furnace plant, will produce 1,300,000 tons of iron per year (an average production of 325,000 tons per furnace).

Stainless Steels in Power Production

The use of stainless steels in the fabrication of machinery for power production—water, steam, and gas turbine—is illustrated in a new 16-mm. colour film produced by Firth-Vickers Stainless Steels Ltd. The film, with a sound commentary, shows how the very stringent demands of modern power production are being met by steels with high resistance to corrosion, erosion, and scaling at high temperatures.

Copies of the film are freely available for use by interested organizations.

Kelvin Medal

The Kelvin Medal for 1953 has been awarded to Dr. Chalmers Jack Mackenzie. Members of The Iron and Steel Institute have been cordially invited to attend the presentation, to be made by Field Marshall The Right Hon. Earl Alexander of Tunis, K.G., at the Institution of Mechanical Engineers, Storey's Gate, London, S.W.1, on 2nd June at 5.30 p.m. No tickets of admission are required.

Percentage Elongation Chart

The inter-conversion of percentage elongations corresponding to different gauge lengths of steel has been simplified by the production of a small ivorine chart by Stewarts and Lloyds Ltd. The data are based on values given in B.S. 806: 1954.

High Duty Alloys Ltd.

High Duty Alloys have taken over three works of Richard Thomas and Baldwins Ltd., as from 2nd January, 1954. They are: The Wern Works, Briton Ferry; the Byass Works, Port Talbot; and the Cwmfelin Press and Fabrication Works, Swansea.

Changes of Name and Address

BRITISH ELECTRO METALLURGICAL CO., LTD., will in future be known as Union Carbide Ltd. The business of manufacturers and suppliers of Ferro Alloys carried on by the Company will in future be carried on under the name of British Electro Metallurgical Co., a Division of Union Carbide Ltd.

The Head Office and Registered Office of the DE HAVILLAND ENGINE CO., LTD., has removed from Edgware to Leavesden Airfield, Leavesden, Herts. (Tel. Garston 2261).

CORRIGENDA

Metallography of Delta-Ferrite. Part 1—Eutectoid Decomposition of Delta-Ferrite

The following correction should be made to the paper by Kehsin Kuo (*J. Iron Steel Inst.*, 1954, vol. 176, Apr., pp. 433–441):

In Fig. 8, the magnification is × 1200

Structural Changes During Annealing of White Cast Irons of High S : Mn Ratios

The following corrections should be made to the paper by A. Hultgren and G. Östberg (*J. Iron Steel Inst.*, 1954, vol. 176, Apr., pp. 351–365):

The caption for Fig. 18*c* should read:

(*c*) iron No. 7, annealed in N_2 for 1 hr. at 1045° C.; quenched

Figures 38 and 58*a* should be turned through 90°, and Fig. 39*b* through 180°.

DIARY

4th May—SOCIETY OF CHEMICAL INDUSTRY (Corrosion Group)—Annual General Meeting, followed by discussion on "*Corrosion and Design*"—Chemical Society, Burlington House, Piccadilly, London, W.1, 6.30 P.M.

6th May—LEEDS METALLURGICAL SOCIETY—Annual General Meeting and Film Night—Chemistry Department, The University, Leeds, 7.15 P.M.

6th–7th May—Conference on Unit Processes of Oxidation—The Hague.

26th–27th May—THE IRON AND STEEL INSTITUTE—Annual General Meeting—4 Grosvenor Gardens, London, S.W.1.

31st May–1st June—NATIONAL PHYSICAL LABORATORY—Symposium on "*Creep and Fracture of Metals (A Review of Recent Theoretical Progress)*"—N.P.L., Teddington, Middlesex.

5th–19th June—THE IRON AND STEEL INSTITUTE—Special Meeting in Sweden.

TRANSLATION SERVICE

(The previous announcement was made in the April, 1954, issue of the *Journal*, p. 449).

TRANSLATIONS AVAILABLE

No. 484 (German). H. G. BRANDT: "Ultrasonic Testing of Round Steel Bars for Internal Defects." (*Stahl und Eisen*, 1953, vol. 73, Dec. 17, pp. 1717–1720).

No. 485 (German). H. SIEGEL: "The Treatment of Liquid Open Hearth Steel with Solid Electric Furnace Slag, and the Metallurgical Processes during Tapping." (*Archiv für das Eisenhüttenwesen*, 1952, vol. 23, Nov.-Dec., pp. 417–425).

TRANSLATIONS IN COURSE OF PREPARATION

(Russian). M. A. SOLUTZEN, L. M. CHEMODANOV, and A. A. KUZIN: "Making Steel Castings from Steel Produced in a Small Convertor." (*Liteinoe Proizvodstvo*, 1953, No. 5, pp. 8–11).

(German). K. MÖHL: "The Sampling of Ores and Slags during Ironmaking." (*Archiv für das Eisenhüttenwesen*, 1954, vol. 25, Jan.-Feb., pp. 33–37).